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FLOTATION OF WEATHERED SILICATES

By

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In recent years the mining industry has turned to by-products to off-set the "profit squeeze" caused by rising manufacturing costs and relatively stable end product prices. This trend to by-product production is noticeable in both metallic and non-metallic or industrial mineral operations. With relatively small capital investments such products as agriculture limestone, road aggregate and fillers have been produced from what was formerly mine or mill wastes.

Several years ago this writer had the opportunity to take an active part in producing salable products from a mica plant tailing. The intent of this paper is to describe the research work performed on the Kings Mountain Mica Company's spiral tails at the North Carolina State Minerals Research Laboratory under the direction of W. T. McDaniel.

Briefly, the ore processed in the mica plant is a weathered granite. Mining is done by self-loading Euclid scrapers. The weathering, caused essentially by ground water, acts first on the sodium feldspar (Albite) which weathers to illite or kaolinite. The potash minerals, microline feldspar and mica decompose under more severe weathering conditions to kaolinite and sericite respectively. Quartz is largely unaffected and iron bearing minerals are oxidized during weathering conditions. During the mica processing the ore is split into three fractions:

(1) A fine clay-like tailings from the washing classifier overflow.

(2) A course sand tailing from the Humphrey's spirals.

(3) The mica product.

Generally, the mica product accounts for only 10 percent of the head feed with the remaining 90 percent being equally divided between the coarse and fine tailings. Previously, research work to market the fine clay tailings had been completed.

The first step in marketing the coarse tailings from the Humphrey spirals was evaluation. It was apparent from the initial assays that the tailings had an economic potential due to the content of high potash feldspar. In addition to the microcline feldspar there was also quartz, a lower grade mica and the cost of tailings disposal for these minerals. It was also apparent in the very early stages of this program that the major problem would be to reduce the iron content of the feldspar concentrates to a commercial level. While some of the initial feldspar concentrates showed 12 to 13% K_2O, they also contained as much as 0.50% Fe_2O_3.

It was also believed that due to the different degrees of weathering in the mica ore body a possibility existed that the mineralogical composition of the spiral tails and the chemical composition of the individual minerals would vary during relatively short time intervals. In order to determine the extent of change a test procedure was set up to measure the uniformity of the spiral tails over a period of several months. Shift samples were collected by an automatic sampler during each month of a four month period. These samples were treated by identical bench scale float tests and screen analyses. Results of these tests were surprisingly uniform. The 19 shift samples evaluated showed an average feldspar concentrate to be 17.3 percent of the head feed and contain 12.6% K_2O and a total alkali content of 13.22 percent. At this point it was concluded that any change in the spiral tails would not be enough to affect the operation of a flotation plant.

Of course, these analyses are quite far apart from the theoretical analysis of potash feldspar. In order to evaluate the flotation process a sample of absolutely pure feldspar was obtained by hand picking. These feldspar particles gave the following analysis:

K_2O = 14.3 percent

Na_2O = 0.46 percent

Fe_2O_3 = 0.025 percent

Using this as a basis for a pure feldspar concentrate these initial tests gave a concentrate of 90 percent grade with flotation recovery in the feldspar step of plus 95 percent. It was decided at this point to settle for a concentrate grade of 12 percent K_2O. This was based on two facts. One being that this was a reasonable goal to expect for a flotation plant and second that this would be the highest potash feldspar commercially available in the United States. This, then would put a new plant in a competitive position without undue demands on the operating conditions.

Further work in the program indicated that the ore had more variation than was predicted by the above bench scale work. The variation was caused by a change in the mining location and occurred mostly in the soda content of the feldspar concentrate. Evaluation of the 19 different shift samples showed the highest percent Na_2O in the feldspar concentrate to be 0.9, whereas in the pilot plant the highest was 2.56 with 1.2 percent Na_2O being common. For example, pilot plant No. 15 produced a feldspar concentrate assaying 11.6 percent K_2O and 2.23 percent Na_2O. In this case, the K_2O content was below 12 percent because of the low K_2O to Na_2O ratio and not because of a low grade concentrate. Listed below are typical $K_2O:Na_2O$ ratios during different phases of the program:

Sample Origin	% K_2O	% Na_2O	Total Alkali	Ratio K_2O:Na_2O
Hand Picked Feldspar grains	14.3	0.46	14.71	31:1
Average of 19 Shift Samples	12.6	0.62	13.22	20:1
Pilot Plant No. 15 Concentrate	11.6	2.23	13.83	5.2:1

These ratios represent the extremes in the chemical composition of the feldspar which was the only variable of any consequence. A system of blending the feed prior to flotation was designed in order to compensate for these changes.

The Fe_2O_3 analysis of the feldspar concentrate at this point was 0.34 percent. The analysis of the pure feldspar grains, only unstained grains were picked, as previously mentioned was only 0.025% Fe_2O_3. This showed that it was possible to meet commercial specifications if enough of the iron bearing minerals and/or iron stained feldspar could be rejected in the iron float. The flotation procedure at this point consisted of five steps:

(1) Scrubbing at 60% solids for five minutes.

(2) Desliming.

(3) Mica flotation with amine as the collector in an acid circuit.

(4) Iron flotation with petroleum sulfonate as a collector in an acid circuit.

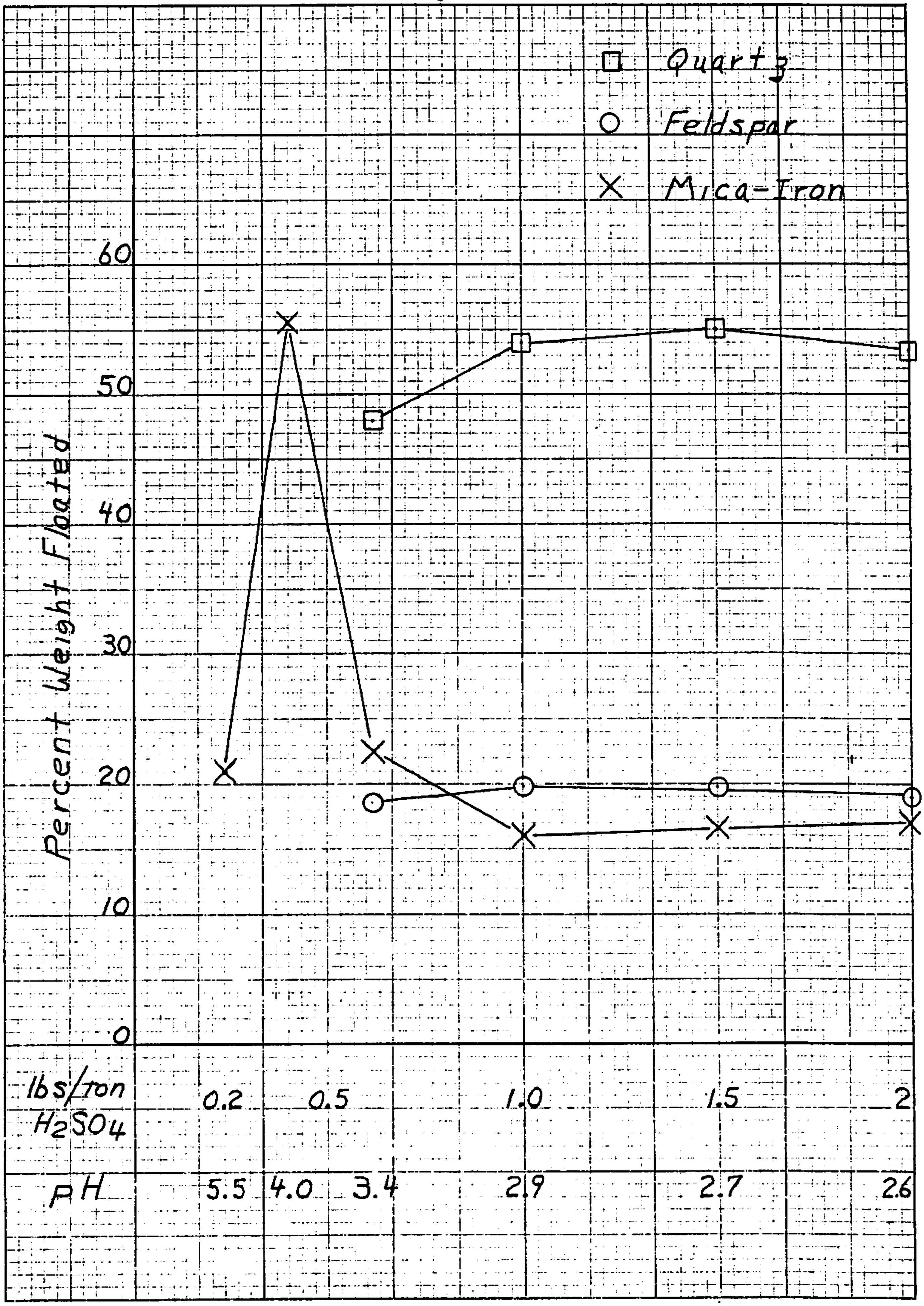
(5) Feldspar flotation with the amine-HF system.

It was noticed that the petroleum sulvonate used as the iron mineral collector also had a marked tendency to act as a collector for mica; probably due to iron activation. Thirty percent of the mica in the head feed was badly iron stained. The mica, both stained and unstained, which did not respond to the mica or iron float reported with the feldspar product which contributed to the high percent Fe_2O_3 in that product.

It was decided that a mica-iron float, i.e., one that would float all of the mica (both stained and unstained) and the iron bearing minerals would be beneficial or even necessary to produce commercial feldspar from this weathered feed. About four months were spent testing variables with petroleum sulfonate as the collector. Among the most important variable tested were pH, soluble iron concentration, quantity of the collector, percent solids of the conditioner and effect of slimes on the collector. Figure #1 is a graph of the percent weight floated versus pH using a constant petroleum sulfonate addition (1.5 lbs. per ton). It can be seen that there is a wide fluctuation over a relatively narrow pH range. At a pH of 5.5 almost no mica was floated and there was excess froth. In the pH range of five to three there was unselective flotation. It is believed this is due to activation by something other than iron. Bhappu (1) states that the aluminum ion can also cause activation near a pH of four depending on the concentration of the ion and of the collector. Since the concentration of the soluble Fe ions in the flotation pulp was lower at a pH of four than at a pH of three and that there is an ample supply of aluminum ions from the feldspar and mica it is reasonable to expect the aluminum ion is responsible for unselective flotation in this pH range. At a pH of below 2.9 or sulphuric acid addition to the condition above 1.0 pounds per ton optinum mica-iron flotation was achieved.

(1) A.I.M.E. transactions of the Society of Mining Engineers Vol. 226, Dec. 1963". The Role of Hydrolysis in Sulfonate Flotation of Quartz by M. C. Fuerstenau, C. C. Martin, & R. B. Bhappu.

Figure 1



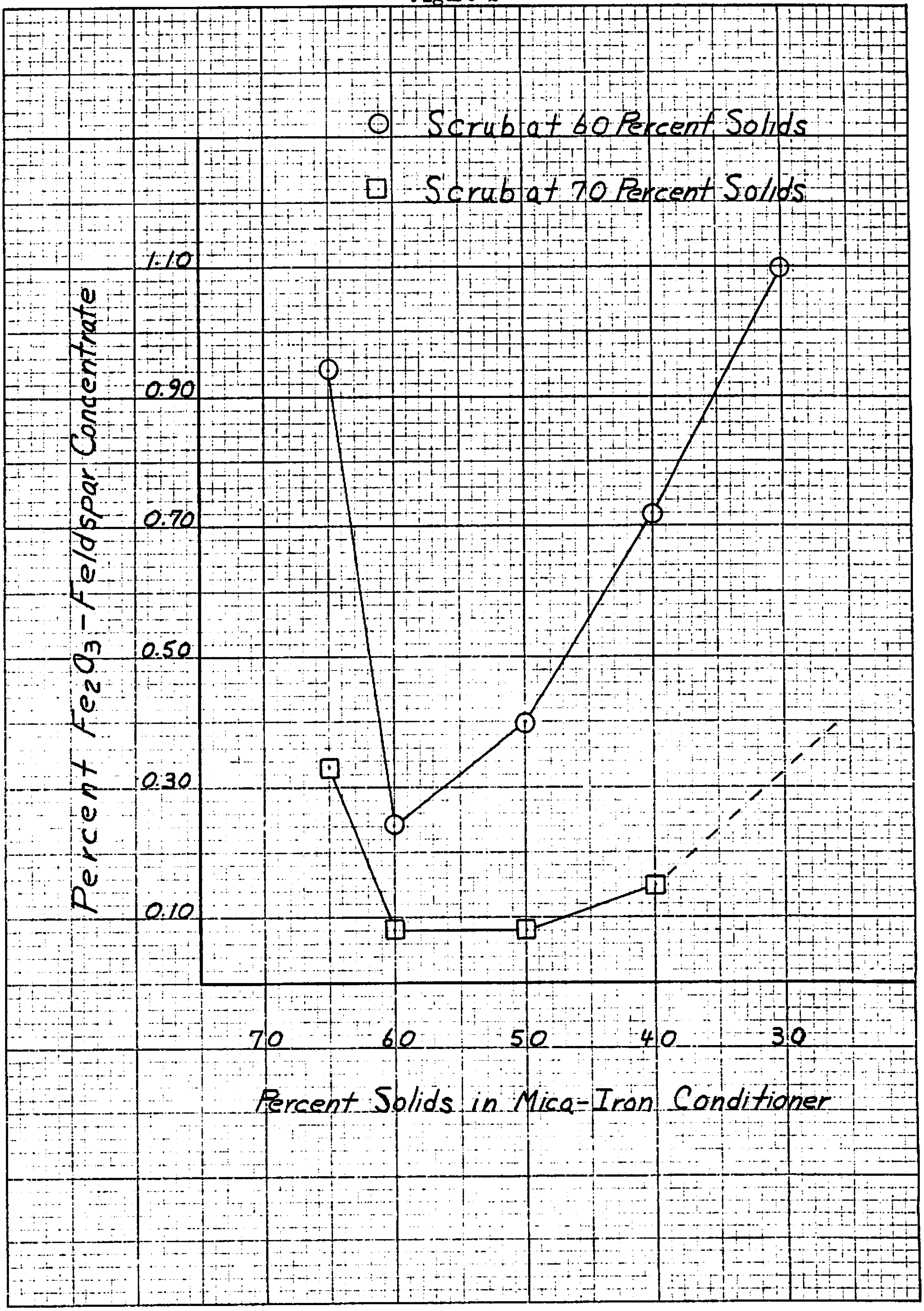
Quartz
Feldspar
Mica-Iron
Percent Weight Floated
60
50
40
30
20
10
0
lbs/ton H_2SO_4
0.2
0.5
1.0
1.5
2
pH
5.5
4.0
3.4
2.9
2.7
2.6

The soluble iron in the mica-iron flotation circuit was in the range of 35 PPM Fe_2O_3. To further study the effect of this ion on the flotation circuit it's concentration was increased with the addition of ferric chloride ($FeCl_3$) and decreased with an addition of a chelating agent, Veresene 100.[2] With the addition of 0.3 pounds per ton of $FeCl_3$ the weight of the mica-iron float product increased from the normal 20 percent to 56 percent. With the addition of 0.5 pounds per ton Veresene 100 the weight floated decreased to 13 percent. With this 35 percent decrease in weight the percent Fe_2O_3 in the feldspar concentrate increased seven fold from 0.1 to 0.7 percent.

Figure 2 shows graphically the results of a series of tests designed to study the effect of the percent solids in the mica-iron (petroleum sulfonate) conditioner. In the upper curve the minus 28 mesh head feed was scrubbed for five minutes at 60 percent solids liberating three percent slimes. In the lower curve the minus 28 mesh head feed was scrubbed for five minutes at 70 percent solids liberating five per cent slimes. It is readily apparent that 60 percent solids in the conditioner gave optimum iron rejection. However, it was far more interesting to note that as the percent solids in the conditioner approached the percent solids in the initial scrub iron rejection deteriorated. This implied that additional slimes were being created in the conditioner. Still more important was the vastly improved results when scrubbing took place at 70 percent solids and that still more slimes could be liberated with additional work input.

(2) The Dow Chemical Co. Essentially a tetrasodium salt of ethylene diamenetetracetic acid.

Figure 2

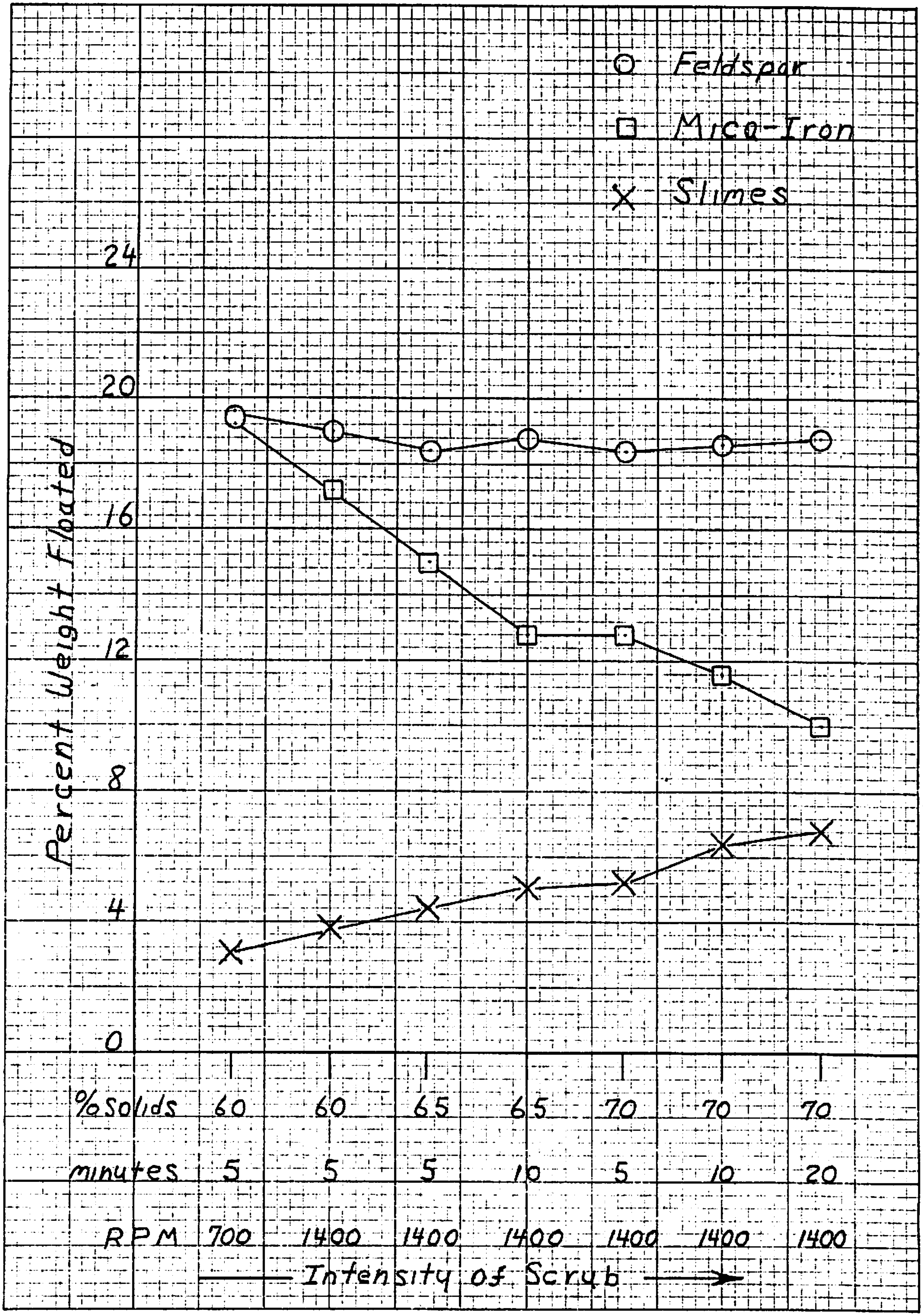

Scrub at 60 Percent Solids
Scrub at 70 Percent Solids
Percent Fe_2O_3 - Feldspar Concentrate
1.10
0.90
0.70
0.50
0.30
0.10
70
60
50
40
30
Percent Solids in Mica-Iron Conditioner

Additional work was immediately done to verify the beneficial effect of high solids scrubbing prior to flotation. Figure 3 shows how increasing the intensity of the scrub several fold affects the weight percent of the different products. Figure 4 gives the percent Fe_2O_3 in the feldspar and quartz products at the different intensities of scrubbing. Following scrubbing, under conditions indicated on the graph, the ore was deslimed and the flotation was performed under identical conditions. The mica-iron flotation conditions are as follows:

	Conditioning	Flotation
Time (Minutes)	5	3
% Solids	60	20
pH	2.7	3.3 (end)
H_2SO_4	1.0 lbs/ton	
Petroleum Sulfonate	1.5 lbs/ton	

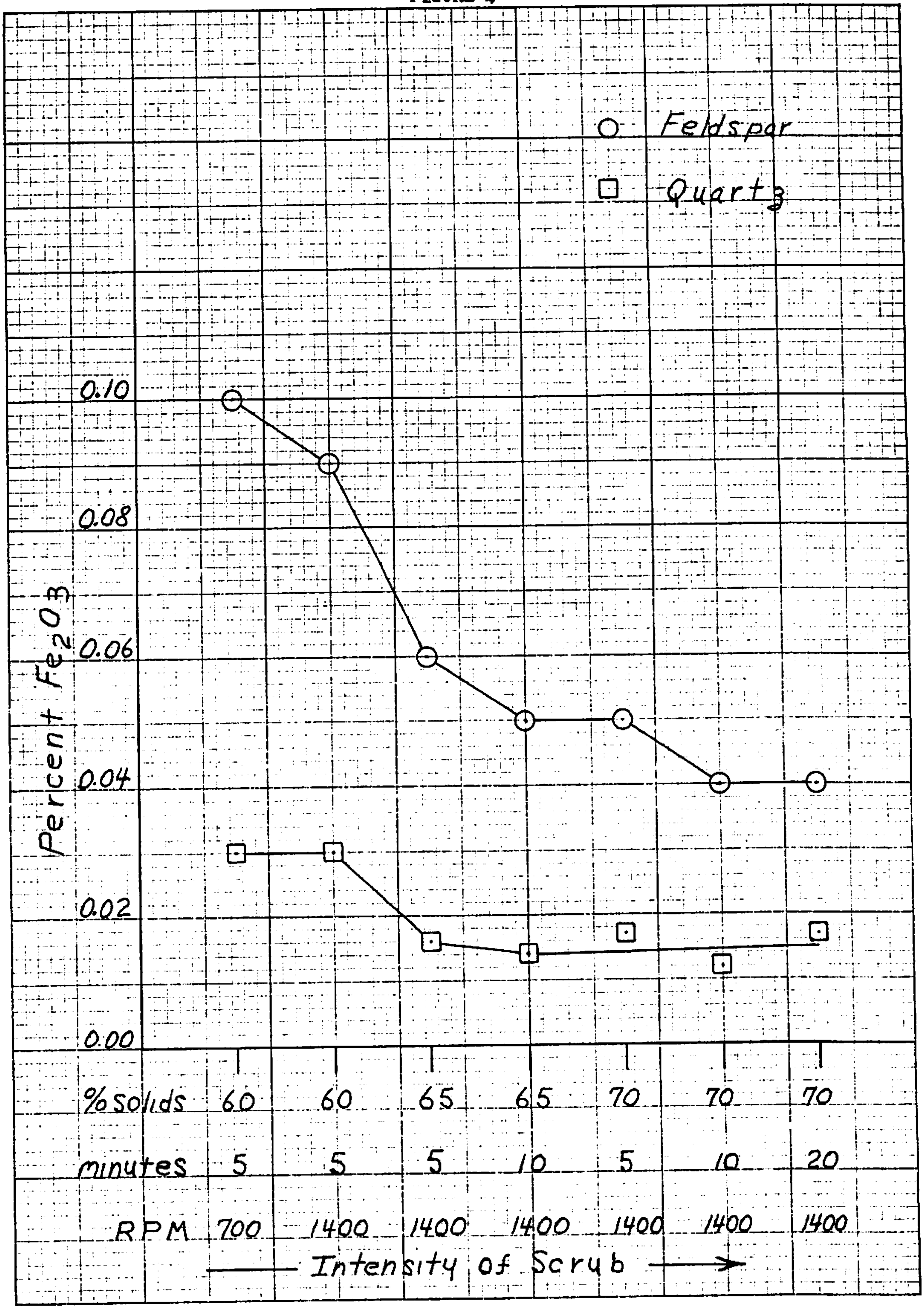
It can be seen from the graphs that as additional slimes are liberated greater selectivity and better iron rejection are obtained in the mica-iron float. For example, the percent Fe_2O_3 in the feldspar product fell from 0.10 percent when scrubbing took place at 60 percent solids for five minutes to 0.04 percent when scrubbing took place at 70 percent solids for 20 minutes. Mica removal was good throughout the series, giving essentially a mica-free feldspar product. On the basis of these results all further test work was scrubbed at 70 percent solids for five minutes prior to flotation. These conditions were chosen because they can readily be duplicated in commercial practice and they produced feldspar and quartz products to glass grade specifications.

Figure 3



Feldspar
Mica-Iron
Slimes
Percent Weight Floated
24
20
16
12
8
4
0
%Solids 60 60 65 65 70 70 70
minutes 5 5 5 10 5 10 20
RPM 700 1400 1400 1400 1400 1400 1400
Intensity of Scrub

FIGURE 4



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The organic nomenclature for petroleum sulfonate is sodium alkyl aryl sulfonate. This reagent is supplied commercially under three trade names:

(1) American Cyanimid's 825.

(2) Mineral Oil Refining Company's Mahogany Soap 62

(3) Shell Oil Company's Sodium Sulfonate Regular.

When used in equal strength with relation to the sulfonate content these three reagents gave very similar results and could be considered equal in both metallurgical performance and cost.

As the above work indicates the feldspar product and especially the iron content of the feldspar concentrate had the highest priority. However, some work was done on each of the other minerals in the feed and on various aspects of the feldspar float before the pilot plant phase of the program was begun.

The quartz product, which is the tailings from the other flotation steps was quite pure throughout the research program. It was always free of mica, contained very little feldspar and was low in iron. Listed below is an analysis of a composite sample obtained from bench scale tests.

% Fe_2O_3	=	0.05
% K_2O	=	0.06
% Na_2O	=	0.03
% Al_2O_3	=	0.14
% Ign. Loss	=	0.29
% SiO_2	=	99.36
		99.93

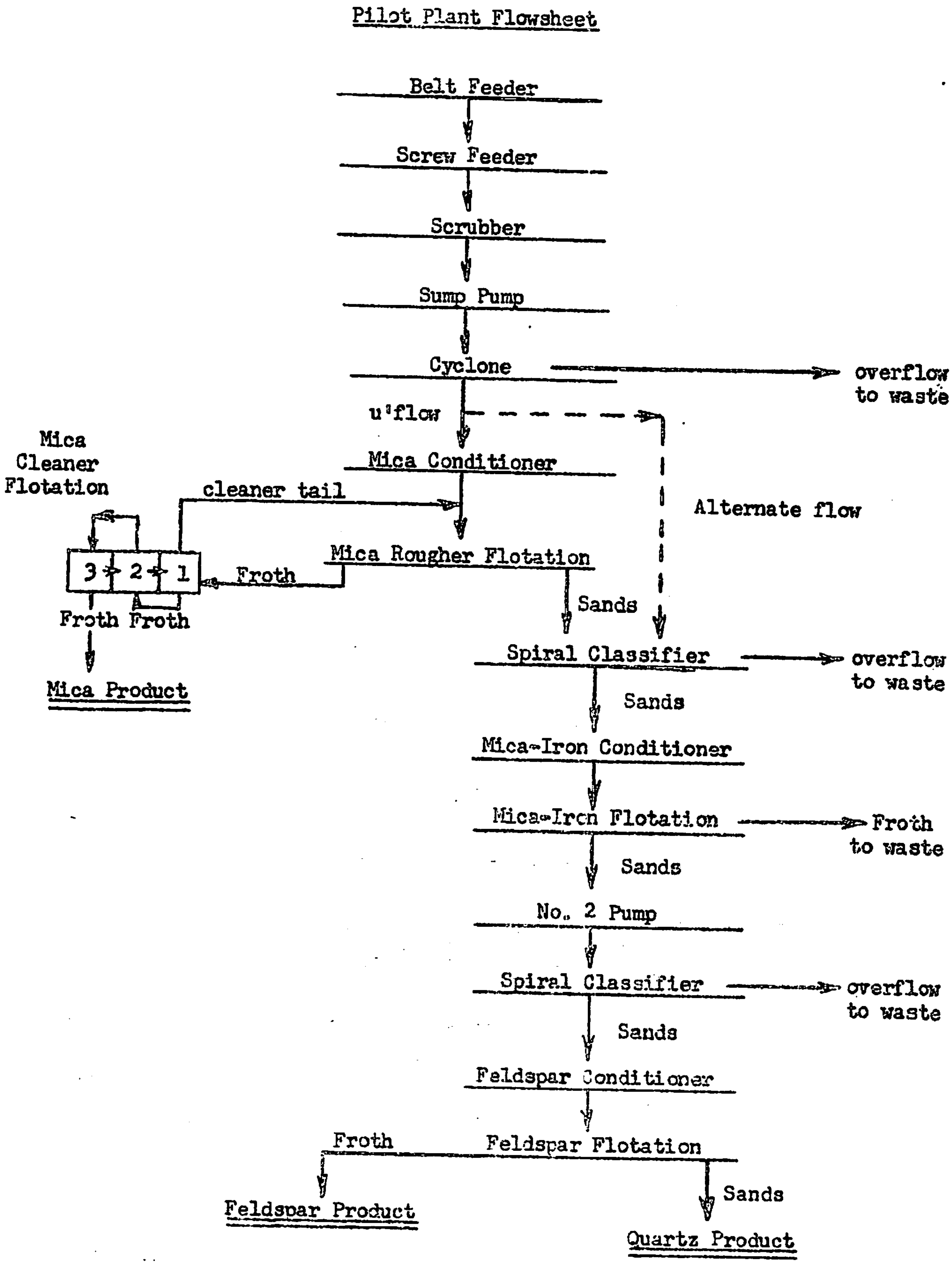
Later, as shown in Figure 4 and verified in the pilot plant, the iron content was lowered to 0.02% Fe_2O_3 by intense scrubbing prior to flotation. Normally, the quartz product contained less than one percent feldspar.

The mica in the head feed was that which was rejected by the spirals in the mica processing. Early in the program it was decided to limit the size of the flotation feed to 28 mesh. While this was primarily to obtain better metallurgy a second advantage was quickly noticed. The plus 28 mesh mica was of good quality and apparently was rejected from the spirals because of its size rather than the amount of delamination. By closing the rod mill with a 28 mesh screen the recovery of primary mica was increased and optimum flotation feed size was obtained. A mineralogical examination showed the flotation feed to contain 7.4 percent mica of which 31 percent was iron stained. During the initial stages of this program the mica was floated in an acid circuit, with amine as the collector. While excellent metallurgy was not achieved grades of close to 90 percent and recoveries of 70 to 80 percent were common. After satisfactory iron removal was reached by intense scrubbing and a mica-iron flotation step the two-step process was again tried. Iron removal in the separate mica and iron scavenger floats was equally satisfactory, but it appeared that the increased scrubbing, which was necessary to produce high quality feldspar and quartz, had a detrimental effect on the mica grade. When preceded by the high intensity scrub, the grade of the mica concentrate dropped to 80 percent or less. About this time a flotation procedure was developed by the U. S. Bureau of Mines [1] and successfully tried in the field. This process also was successful on this ore and was used in the pilot plant phase of this program.

In the fall of 1964 arrangements were made to conduct a pilot plant project on the spiral tails. The object of this project was to verify bench scale test results, obtain data and product samples to determine the economic feasibility of a commercial plant and to obtain metallurgical data for design purposes. Approximately 25 tons of spiral tailings were processed in the pilot plant. The ore was delivered in four-ton lots. Each lot, since it came from a different location in the mica pit was treated separately. Each lot was "made" shortly before being delivered to the laboratory. The procedure consisted of pumping the spiral tails to a 28 mesh Universal vibrating screen. The oversize was returned to the rod mill ahead of the spirals. The undersize was dewatered in two Krebbs cyclones, the underflow being stacked for shipment. The circulating load was built up for one hour before the sample was collected. The feed rate of the pilot plant was determined by pre-weighing the feed into barrels and correcting for moisture. Product rates or material balances were determined by taking timed samples each hour. Chemical analyses were performed on eight hour composites. Figure 5 is a flow sheet of the pilot plant. This discussion of test results is divided into unit operations, with each operation being discussed briefly.

(1) Society of Mining Engineers of A.I.M.E. <u>Selective Flotation of Mica From Pegmatites</u>. James S. Browning & Ralph B. Adair, Tuscaloosa Metallurgy Research Center. U.S. Dept. of Interior, Tuscalloosa, Ala.

FIGURE 5


Pilot Plant Flowsheet
Belt Feeder
Screw Feeder
Scrubber
Sump Pump
Cyclone
overflow to waste
u'flow
Alternate flow
Mica Cleaner Flotation
Mica Conditioner
cleaner tail
3
2
1
Froth
Mica Rougher Flotation
Froth Froth
Sands
Mica Product
Spiral Classifier
overflow to waste
Sands
Mica-Iron Conditioner
Mica-Iron Flotation
Froth to waste
Sands
No. 2 Pump
Spiral Classifier
overflow to waste
Sands
Feldspar Conditioner
Froth
Feldspar Flotation
Sands
Feldspar Product
Quartz Product

Scrubbing: The ore was uniformly fed by a belt feeder into a screw which fed the scrubber. The scrubber was laboratory designed (after Wemco) with two compartments and a total volume of one cubic foot. Each compartment had three four-bladed, reverse pitch, rubber coated impellers of 10-inch diameter. During the initial pilot plant runs, iron rejection was not sufficient to produce glass grade feldspar. It is believed that inadequate scrubbing was the major factor. Before iron specifications were met several variables were changed. The more important ones were horsepower and impeller speed. The horsepower was increased from a total of two to six (two 3 hp motors). At the same time the impeller speed was reduced from 1300 to 950 rpm. Excess speed created a large vortex in the pump, thus reducing retention time and particle contact in the impeller zone. As evidence of this; as the speed was decreased power consumption increased. It was possible to pull full load amperage at constant percent solids by decreasing the speed when the horsepower was increased. The percent solids of the scrubber pulp was always maintained at a minimum of 70 percent solids. From the heat generated in the pulp it was apparent that there was more work input in continuous operation than in bench scale work. It is believed that this was necessary due to short circuiting through the two compartment scrubber. While it was not feasible to correct this in the pilot plant, it was easily corrected in commercial operation. Two other points of interest tested in the pilot plant are scrubber control and dispersants. Pilot plant tests showed that 1.0 pound per ton of NaOH increased the percent weight of the slime fraction from 4.8 to 7.2 percent which, in turn, resulted in a more selective mica-iron float. (see Figure 3). The scrubber was controlled, very satisfactorily, by monitoring the amperage of the number one scrubber motor.

It was recommended that this be automated in commercial practice.

Desliming: The scrubber discharge was pumped in a dilute pulp (5 percent solids) to a cyclone for primary desliming. An excellent separation was made in the 3-inch Dorrclone used. The cyclone overflow accounted for 5.9 percent of the head feed of which only 0.4 percent was plus 325 mesh material. A secondary desliming operation was carried out in a Denver spiral classifier. This was chosen rather than two-stage desliming to present a more uniform percent solids to the mica-iron conditioner. The total slime loss throughout the pilot plant was uniform averaging 6.5 percent of the head feed.

Mica Flotation: In the pilot plant the mica was treated in either of two ways: (1) It was floated in a mica-iron float as a waste product. (2) It was floated separately to recover mica as a salable product. In the second method the mica not recovered in the mica float was floated in the mica-iron scavenger float to prevent contamination of the feldspar product. The flotation process used to recover the mica was a cationic-anionic float originally developed by the Bureau of Mines of the Tuscaloosa Alabama Station. In the initial runs, not enough conditioning and/or roughing capacity was installed. This was changed by installing a 1.2 cubic foot Denver Super conditioner, four No. 8 Galigher cells as roughers and three No. 5 Denver cells as cleaners. In these runs good mica grade and recovery were achieved. The average of pilot plant runs 23, 24 and 25 show the mica cleaner concentrate as 5.0 percent of the head feed, assaying 92 percent grade with 93 percent mica recovery.

Mica-Iron Flotation: The mica-iron step was designed to float all of the mica and iron-bearing minerals (mica-iron) or the iron bearing minerals and that mica which was not recovered in the mica float (mica-iron scavenger). In either case, the reagent requirements (petroleum sulfonate and sulphuric acid) were the same. Flotation was carried out in a four-cell bank of No. 8 Galighers, with a junction box between the second and third cells. This float had been extensively tested in bench scale and continuous operation confirmed bench-scale findings. The froth product of the mica-iron step averaged 12.4 percent of the feed and was wasted. Slightly more petroleum sulfonate was required than predicted by bench-scale work. It was believed this was due to the aforementioned scrubbing variables. In one run the percent solids of the mica-iron float was raised from 23 to 32 percent. This increased both the retention time and the reagent concentration in the cells. The petroleum sulfonate was kept constant at 1.6 pounds per ton. However, iron rejection remained low indicating flotation volume was not the factor. Iron rejection was sufficient to produce glass-grade products when the collector was raised to 1.8 to 2.0 pounds per ton.

Feldspar Flotation: The relatively pure feldspar and quartz mixture was separated in a third flotation step. The reagents used were amine, hydrofluoric acid and a frother. Few variables were run on this float during the pilot plant. Good grades and recovery were consistently obtained. One variable tested was conditioning. Three conditioning variables were tested:

(1) None. (No conditioning or dewatering prior to flotation)

(2) Conditioning at 40 percent solids

(3) Conditioning at 60 percent solids.

Feldspar Concentrates - Complete Analysis

	PP-25-A	PP-11*	No. 1 Spar Comp.	No. 2* Spar Comp.
% K_2O	12.8	12.9	11.7	12.1
% N_2O	1.30	0.90	1.51	0.77
% Al_2O_3	18.1	17.9	18.0	16.8
Total Alkali + Alumina	32.2	31.7	31.2	29.7
% SiO_2	66.9	66.9	68.3	69.5
% Fe_2O_3	0.058	0.056	0.064	0.067
% CaO	tr.	tr.	tr.	tr.
% MgO	tr.	tr.	tr.	tr.
Ig. Loss	0.38	0.47	0.34	0.50

* Analyses after magnetic separation.

Feldspar Concentrates - Pilot Plant #13

Size - Chemical Analyses

Tyler Mesh	% Weight	Cum. % Wt.	% K_2O	% Na_2O	% Fe_2O_3
+28	0.3	0.3	-	-	-
-28+35	0.7	1.0	12.8	0.69	0.37
-35+48	6.0	7.0	12.2	1.29	0.11
-48+65	17.0	24.0	11.7	1.82	0.085
-65+100	27.2	51.2	11.1	2.50	0.072
-100+150	24.1	75.3	10.4	2.90	0.062
-150+200	12.6	87.9	10.7	2.67	0.059
-200+270	6.5	94.4	11.6	2.28	0.061
-270+325	3.3	97.7	11.5	2.09	0.064
-325	2.3	2.3	11.3	1.80	0.16
Total	100.0	100.0	11.2	2.42	0.065

There were some discrepancies in the test results but there appeared to be a tendency for conditioning at 40 to 50 percent solids to produce a higher grade feldspar concentrate.

Cell Speed: The speed of the Galigher cells as received was 1500 rpm or a peripheral speed (S) of 1650 feet per minute. This produced a great deal of turbulence in the cells and the speed was lowered to 1000 rpm (S = 1100). The Galigher Company recommends a peripheral speed of between 900 to 1320 feet per minute, depending on cell size.

Feed Rate: The feed rate to the pilot plant was varied from 248 to 468 pounds per hour. The design feed rate, used in the majority of tests, ranged from 330 to 350 pounds per hour. This range was used to scale up to a commercial plant of 400 tpd, giving a scale-up factor of 100.

The table on the following page gives complete chemical and size analysis of several feldspar concentrates produced in the pilot plant.

As a direct result of this research project and especially the pilot-plant phase of the project, a commercial flotation plant was built by the Kings Mountain Mica Company. With certain modifications, the plant was built with a scale-up ratio of 100 to 1 from the pilot-plant data. Plant performance has been satisfactory and has generally substantiated both bench-scale and pilot-plant results.

Acknowledgments

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