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HIGH QUALITY WET GROUND MICA FROM MICA SCHIST ORES

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INTRODUCTION

Chemically, mica is a complex silicate of sodium, potassium and aluminum. There are no important uses of mica where the chemical analysis is important, except that in some uses of ground mica, the content of iron oxide present as an impurity must be kept to a very low limit. Muscovite mica is frequently found containing black and red "stain" as a homogeneous part of the books or sheets. In most cases this stain or impurity is magnetite or hematite. Some mica contains impurities deposited by infiltration. This is commonly a red stain, less often brown or white. The best mica is free from all such impurities, and the greater their content, the less value does the mica have.(1) It is because of these undesirable impurities that a large segment of the fine ground mica industry relies on weathered pegmatites and alaskite ores as a source of raw material. The mica concentrates obtained from these ores are relatively clean and require moderate beneficiation to meet color and density specifications. One need only to observe the stained and decrepitated condition of the mica recovered from mica schist to appreciate the challenge that processing this material presents. Despite these adversities there are several advantages to be gained by mining and processing mica schist ores. The mica content of these ores are considerably higher than the pegmatites and alaskite ores now being mined, often containing in excess of forty percent mica. This higher grade mica feed should promote a lower mining and milling cost with its resulting competative advantage. With more rigid government

regulations and closer scrutiny, the storage and disposal of tailings are becoming a considerable problem. The mica schist ores with their higher mica content would mean less tailings to handle. With these thoughts in mind, a flowsheet was developed whereby mica could be recovered from mica schist ores and beneficiated in a way that would result in a high quality end product.

SUMMARY

The Laboratory batch tests have demonstrated the technical feasibility of producing wet ground mica from mica schist ores with the same specifications as those now on the market. Approximately 20-25 percent of the ore is recovered as a wet ground mica product with a mica recovery of 50-60 percent. No attempt was made to recover a lower grade mica product which would increase the over all recovery.

DEVELOPMENT OF FLOWSHEET

While developing a flowsheet for the beneficiation of mica schist ores, several phases of the flowsheet required particular attention. These will be elaborated on, not in the order of their development, but in the order in which they occur in the flowsheet.

Flotation

The mica is concentrated by flotation using either of two methods: an amine float in an acid circuit, or the Bureau of Mines amine-fatty acid float in a basic circuit. The writer does not wish to elaborate on the relative merits of the two flotation procedures; however, after investigating ores from various localities, it was felt that the amine float in an acid circuit was more consistent when using a standard procedure.

Iron Reduction and Magnetic Separation

This phase of the flowsheet may be considered the key to the process. It was developed when it became apparent that hot acid leaching had just about reached its maximum effectiveness for color improvement. Professor S. C. Sun and William Hirsh in their publication, "Hydrochloric Acid Leaching of Iron From Pennsylvania Aluminous Clays," reported iron extraction for minus

20 mesh minerals of 82.20 percent for hematite and 33.46 percent for magnetite. The test procedure included a 19 percent hydrochloric acid solution, tested at 104° c. temperature and 10 percent pulp density.(2) It was felt that a pre-leaching stage to remove some of the iron contaminate would therefore be advantageous. Since the iron minerals present as contaminates vary in magnetic susceptibility, it was decided to experiment with reducing reagents as a means of influencing their magnetic properties. Zinc hydrosulfite or sodium hydrosulfite were the reagents used throughout the tests. The procedure developed consists of conditioning the mica concentrate in a closed container at approximately five percent solids with a reducing reagent. The ferric iron is reduced to the ferrous state (magnetic) and oxidation retarded by being isolated from the atmosphere in a closed container. After the desired conditioning time has elasped, the material is passed through a Frantz Ferrofilter where the magnetic particles are retained and discarded as waste. The non-magnetic material is recovered and prepared for leaching. The benefits obtained by including this stage in the flow sheet have been verified by processing a number of mica schist ores. While there is no doubt about the effectiveness of the procedure, there did remain a question as to

whether a magnetic alteration was being developed or whether the improvement resulted entirely from converting the ferric iron to a soluble ferrous iron.

In order to resolve this question, a series of tests were run under closely controlled conditions using identical feed samples. These tests consisted of:

1. Conditioning with zinc hydrosulfite followed by wet magnetic separation using an Eriez High Intensity (20 amperes) Separator.
2. Wet magnetic separation using an Eriez High Intensity Separator.
3. Conditioning with zinc hydrosulfite followed by wet magnetic separation using a Frantz Ferrofilter (1 ampere).
4. Wet magnetic separation using a Frantz Ferrofilter (1 ampere).
5. Wet magnetic separation using a Frantz Ferrofilter (1 ampere) followed by conditioning with zinc hydrosulfite.
6. Conditioning with zinc hydrosulfite only.
7. Acid leaching only.

Each test was followed by the standard acid leach and grind. In the tests using the Eriez magnet, the magnetic product was increased by 40 percent after adding a reducing reagent prior to magnetic separation. In the tests using the Frantz Ferrofilter, the magnetic product was increased by 170 percent after adding a reducing reagent prior to magnetic separation. A comparison was made of the final products colors

as determined by a Photovolt Reflectance Meter, the colors being indicative of the amount of iron remaining in the samples. The samples conditioned with the reducing reagent prior to magnetic separation resulted in the best colors. The samples that were not treated with the reagent but subjected to a magnetic separation were next in line. The test in which the sample was run through the ferrofilter then conditioned with the reagent showed some improvement over the acid leaching sample. The sample that was conditioned with the reagent and did not receive magnetic treatment showed a slight improvement over acid leaching The results of these tests are shown in Table 1. An additional test was conducted to substantiate the magnetic alteration theory. A sample of mica concentrate was fed to a Frantz Ferrofilter. The magnetic fraction was saved and the non-magnetic fraction fed back through the ferrofilter. The procedure was repeated until three passes had been made through the ferrofilter. The non-magnetics from the third pass was conditioned with a reducing reagent and then fed to the Frantz Ferrofilter. The weight percent magnetics increased markedly from the previous pass and exceeded the first pass slightly (See Table 2).

Leaching

Some mica producers have expressed objections to leaching because of the high cost of consumed acid. Their objections may be valid when the cost is figured on a one time use basis. A series of tests were performed whereby the acid filtrate was reclaimed, increased to its original volume by the addition of fresh acid and re-used for subsequent leaching. No loss in leaching effectiveness occured by recycling the acid.

Grind (Leached Concentrate)

There are several established methods of wet-grinding mica now being used by the industry. While not attempting to discredit these methods, several types of grinding machines were tried under various conditions in an effort to assimilate a marketed product. The pebble mill, using ½ inch alumina balls as a grinding media, was found to be very effective in producing the desired grind. The high solids (65 percent) grind is sufficient to coat the balls with the mica slurry and allow the desired shearing action created by the ½ inch diameter balls due to their small nip angle. The pebble mill grind was standardized by grinding a mica concentrate, furnished by a mica producer, in a pebble mill until it met his product specifications. The grinding time, charge, mill speed, etc. were noted.

PRODUCT SPECIFICATIONS

Because of the lack of firm specifications for mica, which are usually worked out between seller and purchaser, the density and colors of a minus 325 mesh, marketed mica product are used as a standard. The standard is as follows:

	Photovolt Reflectance Reading		
Density lbs per. cu. ft.	Green	Amber	Blue
12.8	74	75	66

ANALYTICAL CONTROL

The percent mica assays for the flotation products are determined with the aide of a Frantz Isodynamic Magnetic Separator. The bulk density of the products is obtained with a Scott Volumeter.

The color readings are determined with a Photovolt Reflectance Meter using green, amber, and blue filters. The readings are taken as a percentage of that of MgO which is assigned the value of 100.

GENERAL PROCEDURE

The mica is floated by either of two methods, an amine float in an acid circuit or the Bureau of Mines amine-fatty acid float in a basic circuit. The flotation mica is ground in a pebble mill to liberate contaminated particles and expose iron stained surfaces. The concentrate is then conditioned with a reducing reagent and passed through a Frantz Ferrofilter for the removal of magnetic material. The non-magnetic product is given an acid leach to further reduce the iron contaminate. The leached material is then ground to product size and checked for specifications.

DETAILED PROCEDURE

Sample Preparation

A representative head feed sample is obtained for future reference and optical analysis. The remaining material is then given a size reduction with a jaw crusher followed by a roll crusher. The ore is divided into five hundred gram samples for batch testing.

Flotation

Grind 500 gram sample four minutes at 25 percent solids in a rod mill with 10 rods and 1.0 pounds per ton of NaOH. Screen rod mill discharge to obtain plus 28 mesh mica product. Deslime minus 28 mesh two times on 325 mesh. Scrub 10 minutes at 65 percent

solids with 2.0 pounds per ton NaOH. Deslime two times on 325 mesh. Condition three minutes at 45 percent solids with either of the following sets of reagents:

Basic Circuit	Acid Circuit
3.4 lbs./ton Goulac	2.0 lbs./ton H_2SO_4
0.5 lbs./ton D.L.R.	1.5 lbs./ton Fuel Oil

Transfer material to flotation cell and condition one minute at 25 percent solids with:

0.2 pounds per ton Armac-T (amine acetate) or 0.5 lbs./ton for acid circuit.

0.25 pounds per ton MIBC (frother)

Float mica, then clean one time after adding 1.00 pound per ton of H_2SO_4 (when using acid circuit). Combine plus 28 mesh mica (screened out before flotation) with flotation mica (approximately 150 grams).

Grind (Flotation Concentrate)

The flotation mica concentrate and plus 28 mesh mica transfered to a pebble mill with 4,000 grams of one-half inch alumina balls (one-half mill volume). The material is ground for 15 minutes at 65 percent solids. The ground mica is transfered to a bucket, water added to constitute a full bucket and allowed to stand for one minute then decant on a 325 mesh screen.

Wet Magnetic Separation

Mica concentrate transfered to a 500 ML polyethelene bottle. Fill bottle up to the neck with hot (115°F.) water, then add zinc hydrosulfite based on (10.0 pounds per ton of mica concentrate). The bottle is capped with a lid in which a hole had been drilled to allow for close fit of the shaft of a glass stirring rod. The concentrate is conditioned for one hour while being stirred continuously. The contents are fed to a Frantz Ferrofilter over a time interval of 5 minutes. The magnetics are rejected as waste and the non-magnetic product retained for leaching.

Leaching

Approximately 150 grams of mica leached in 1000 ML beaker at 25 percent solids with 10 percent H_2SO_4 and 90 to 95°c. for one hour. Stir continuously with mechanical stirrer. Filter hot on Buchner Filter using No. 4 filter paper. Spray wash twice with 250 ML of water. Return to neutral pH by gravity washing for five minutes with 500 ML of water and 10 ML of two and one half percent NaOH.

Grind (Leached Concentrate)

Approximately 150 grams of leached mica ground in pebble mill at 60 rpm for 45 minutes at 65 percent solids with 4,000 grams of one-half inch alumina balls (one-half mill volume) and 10 pounds per ton (based on flotation head feed) of tetrasodium pyrophosphate. Settle mill discharge in full bucket of water for one hour. Siphon off water and suspended solids. These suspended solids contain clay,

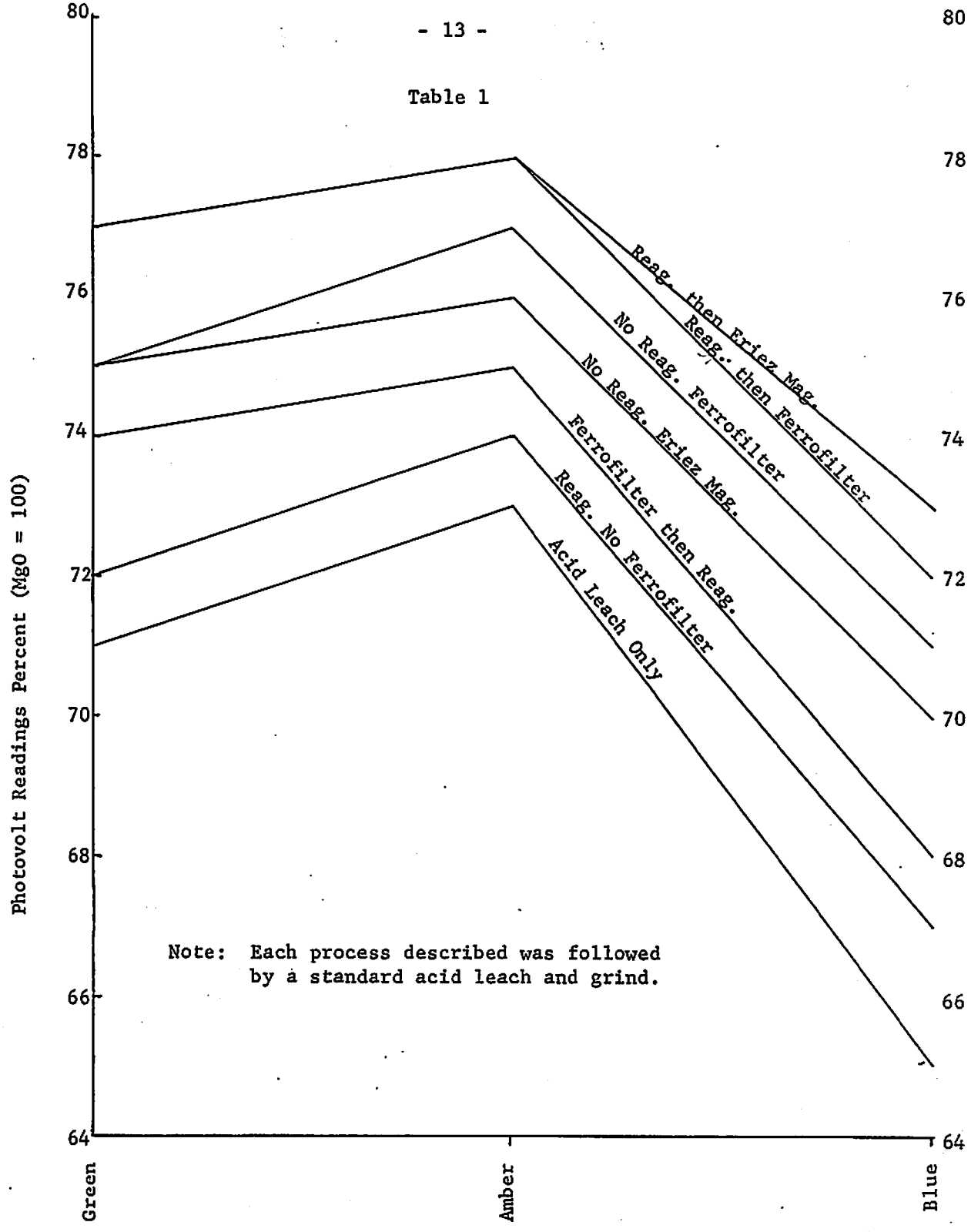
iron oxides and altered mica and they are considered to be waste. Dry settled mica, weigh and calculate grinding recovery assuming no loss in mica will occur in further grinding of oversize. Screen settled mica on 325 mesh and return oversize to pebble mill for additional 1½ hour grind at 65 percent solids without reagents. Dry all of mill discharge and screen on 325 mesh. Combine minus 325 mesh fractions from both grinds, obtain colors and density and record as finished product specifications. Percent mica recovery is recorded by taking into account the flotation, ferrofilter, leaching and grinding recoveries. The yield is recorded as weight of product recovered expressed as percent of ore.

REFERENCES

1. Industrial Minerals and Rocks. Seeley W. Mudd Series, Third Edition. - S. A. Montague: Mica p. 551.

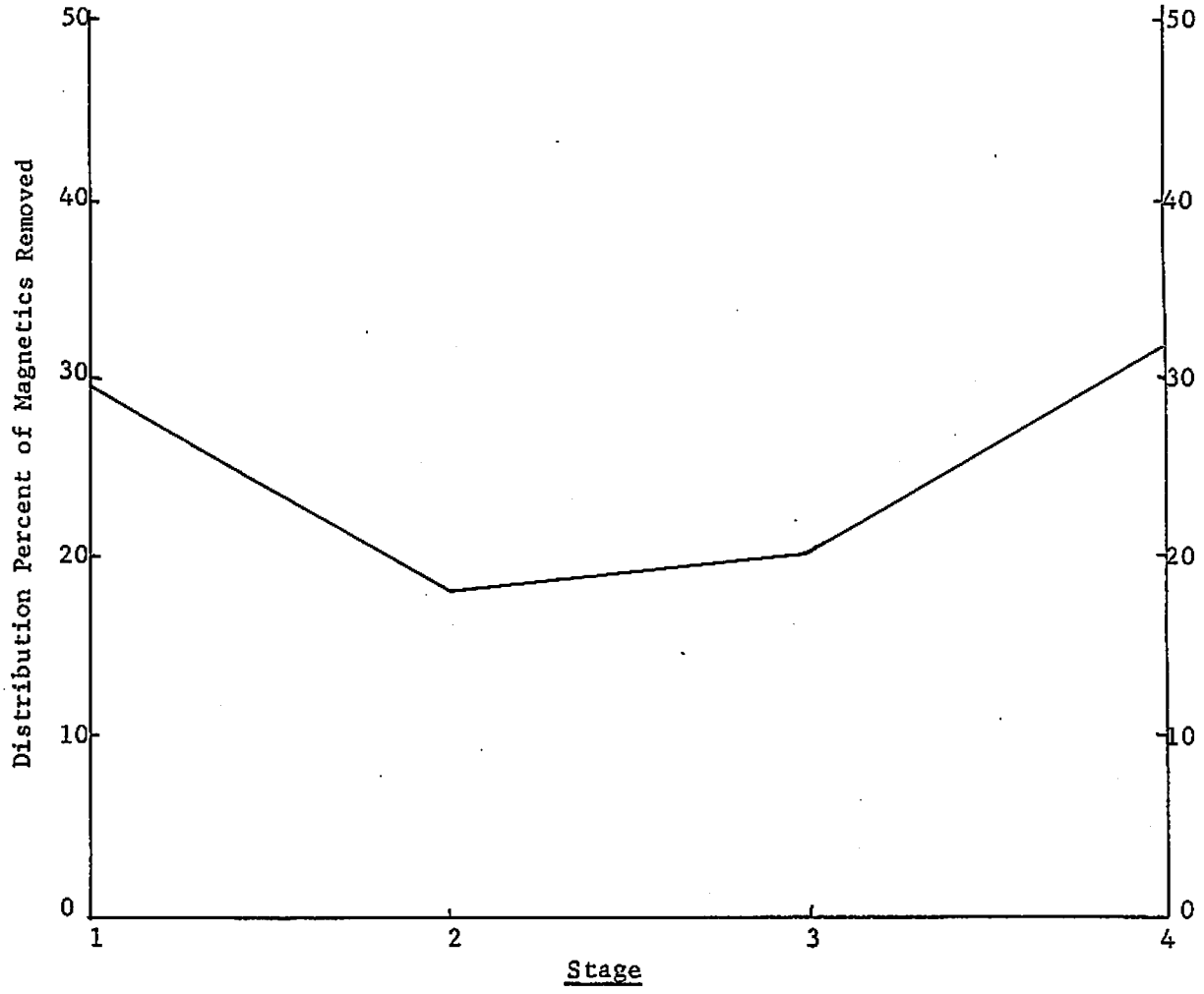
2. Hydrochloric Acid Leaching of Iron From Pennsylvania Aluminous Clays. S. C. Sun, Professor, the Pennsylvania State University, University Park, Pennsylvania. - William Hirsh, Engineer, Abbott Laboratories, Chicago, Illinois.- Presented at the Annual Meeting of the American Institute of Mining, Metallurgical and Petroleum Engineers, New York, New York, February 25-29, 1968.

Table 1



PHOTOVOLT READINGS
OF MICA PRODUCTS
USING VARIOUS PROCEDURES

Table 2



Stage No.

1	First Pass Head Feed.
2	Non-Magnetic product from stage 1 used as feed.
3	Non-Magnetic product from stage 2 used as feed.
4	Non-Magnetic product from stage 3 conditioned with reducing reagent then fed to magnet.

GRAPH SHOWING
INCREASE IN MAGNETICS
WITH ADDITION OF
REDUCING REAGENT

Table 3

Comparative Data For Flowsheet
Without Ferrofilter and With Ferrofilter

Ore Lab. No.	Grade % Mica	Photovolt Reflectance Readings					
		Without Ferrofilter			With Ferrofilter		
		Green	Amber	Blue	Green	Amber	Blue
2026-A	45.2	65	67	59	71	73	64
2026-B	43.6	70	73	64	73	75	67
2033	49.4	70	73	71	76	78	74
2036	41.7	60	63	51	70	71	69
2077	59.7	68	70	67	70	72	68
3079	45.1	75	75	68	78	80	75
3094	41.9	66	69	56	73	75	66

Table 4

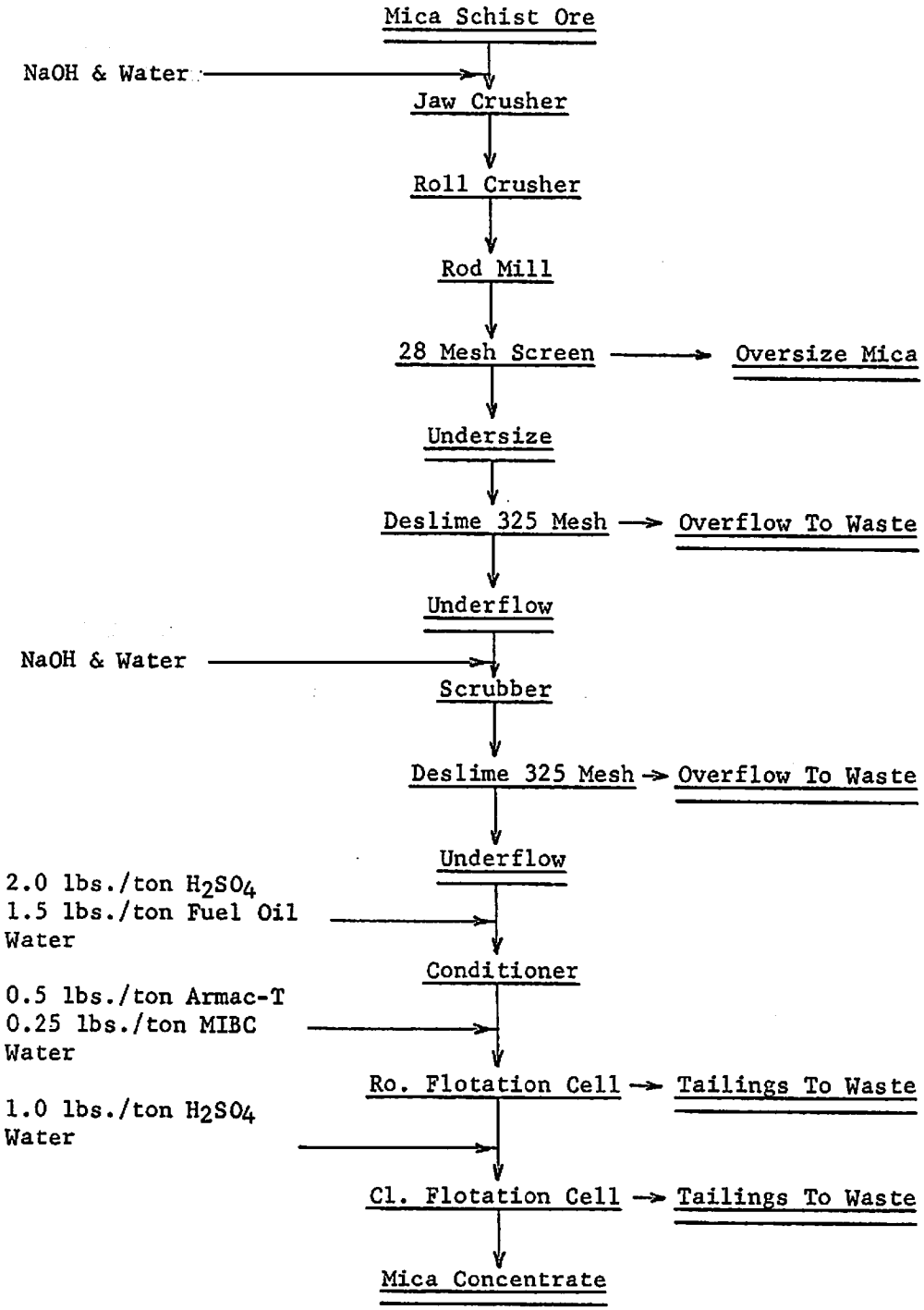


Figure 1. - Flowsheet for Recovery of Mica from Mica Schist Ores.

Table 5

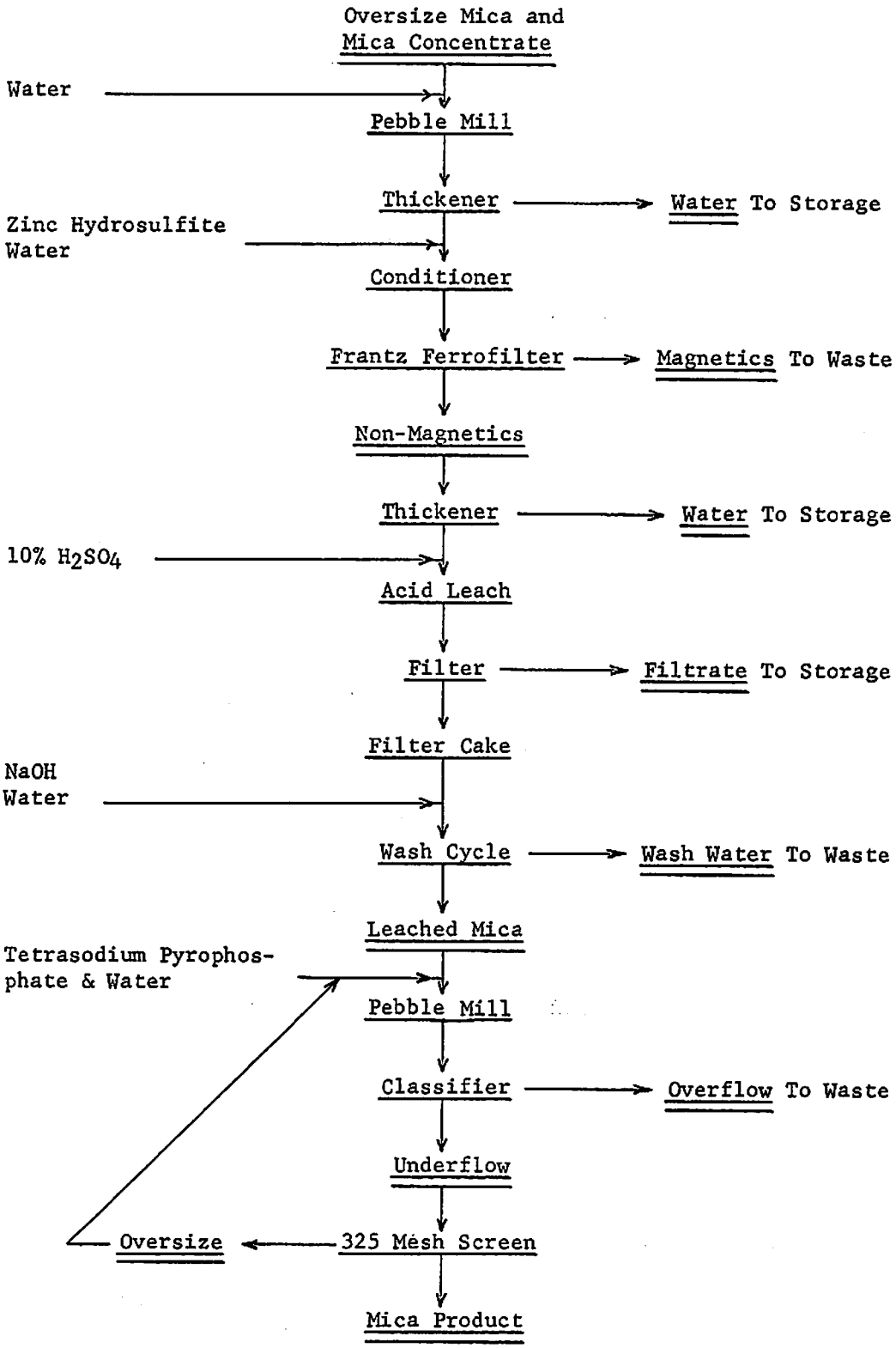


Figure 2. - Flowsheet for Production of Wet Ground Mica.

N.C. STATE UNIVERSITY MINERALS RESEARCH LABORATORY
Mica Schist
ORE DRESSING DATA

Date 8 - 14 - 68 Ore 3079 (comp.)

Engineer _____ Sample No. 70

FLOTATION

Product	Sample Weights			Assays					Mica		
				%	Dens.	Color with filter					
	Grams	Wt.%	Cum.%	Mica	#/cu.'	Green	Amber	Blue	Units	Dist.	Yield
+28	4.2	0.8		100.0					0.80	2.0	
Mica Conc. (F.P.)	167.3	33.5		100.0	42.9	41	41	33	33.50	81.7	
Cl. Mids (M.D.)	59.3	11.9		46.3					5.51		
Ro. Tails (M.D.)	96.0	19.2		6.2					1.19		
#1 Slime (-325)	94.1	18.8		-					-		
#2 Slime (-325)	76.5	15.3		-					-		
Losses	2.6	0.5		-					-		
Total	500.0	100.0		41.0					41.00	83.7	34.3

Process						Reagents (lbs. per ton of feed)						
Equipment	Feed	Time	Solids	pH	rpm	NaOH	H_2SO_4	F.O.	Ar.-T	MIBC		
Rod Mill	500 g.	4	25			1.0						
Screen												
No. 1 Deslime	2x	1										
Scrub		10	75		1750	2.0						
No. 2 Deslime	2x	1										
Mica Cond.		3	45	3.0	700		2.0	1.5				
Mica Float		4.5	18	4.1	1200				0.5	0.25		
Mica Cleaner		4.5	18	3.2	1200		1.0					

Wet Magnetic Separation:		Cum. Mica Dist.	Cum. Mica Yield
Mag.	5.7 %		
Non Mag.	94.3 %	78.9	32.3
Feed	100.0 %		

ACID LEACH OF FLOTATION MICA CONCENTRATE

Product	Weights		Assays				Cum. Mica	
			Dens.	Color w/filter				
	Grams	Dist.	#/cu.'	Green	Amber	Blue	Dist.	Yield
After leach	106.5	91.5	25.1	58	59	50	72.2	29.6
Loss	10.0	8.5						
Before leach	116.5	100.0						

GRIND OF LEACHED MICA CONC.

Product	Weights	
	Grams	Dist.
After grind	76.0	71.9
Loss	29.7	28.1
Before grind	105.7	100.0

FINAL MICA PRODUCT

Dens.	Color with filter			Cum. Mica	
#/cu.'	Green	Amber	Blue	Dist.	Yield
14.3	78	80	75	51.9	21.3

Note: Dist. - Percent of head feed mica recovered in product.
Yield - Weight of product recovered expressed as percent of ore.

Table 7

Summary Data

Distribution and Grades

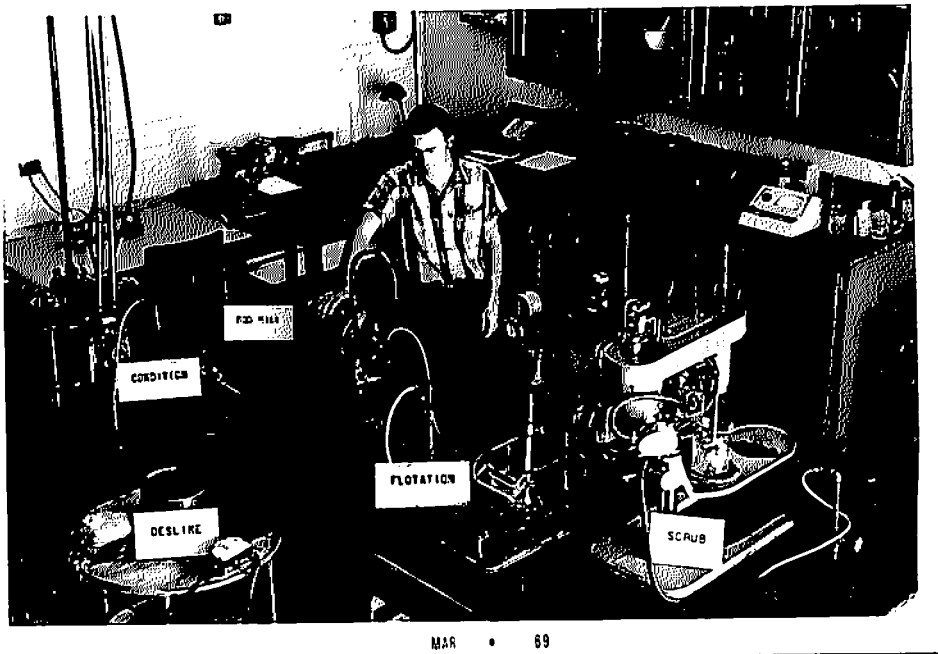
Sample	Wt. %	Mica %
Flotation Feed	65.4	62.7
Slimes (-325 M.)	34.6	-
Head Feed	100.0	41.0

Recovery and Yield

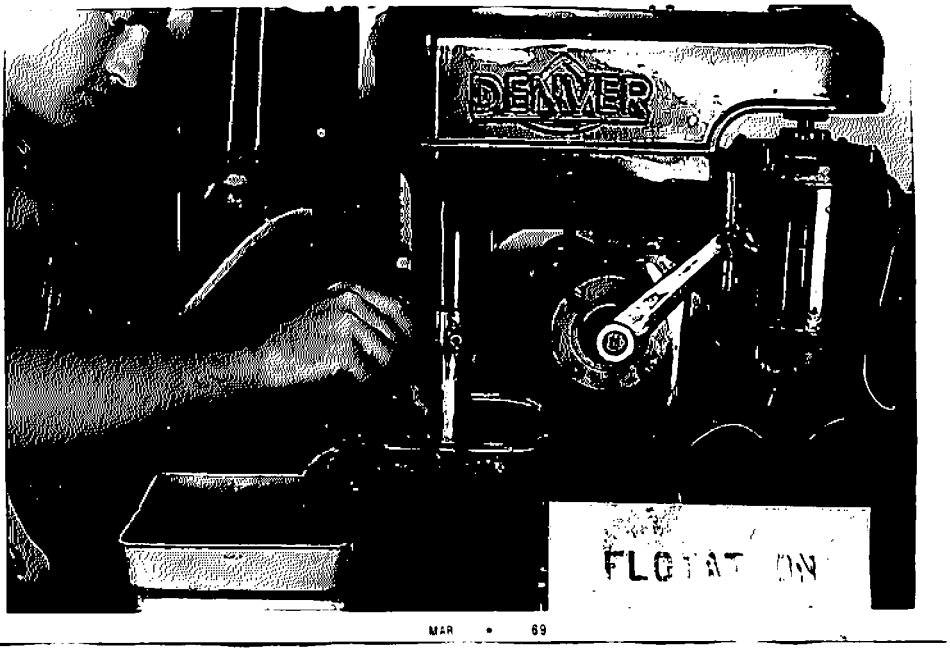
Process	Mica Recovery		Yield
	Unit %	Cum. %	Cum. %
Flotation	83.7	83.7	34.3
Wet Mag. Sep.	94.3	78.9	32.3
Acid Leaching	91.5	72.2	29.6
Grinding (Product)	71.9	51.9	21.3

Note: Mica Rec. - Percent of head feed mica recovered.
Yield - Weight of mica recovered expressed as percent of ore.

Process Photographs

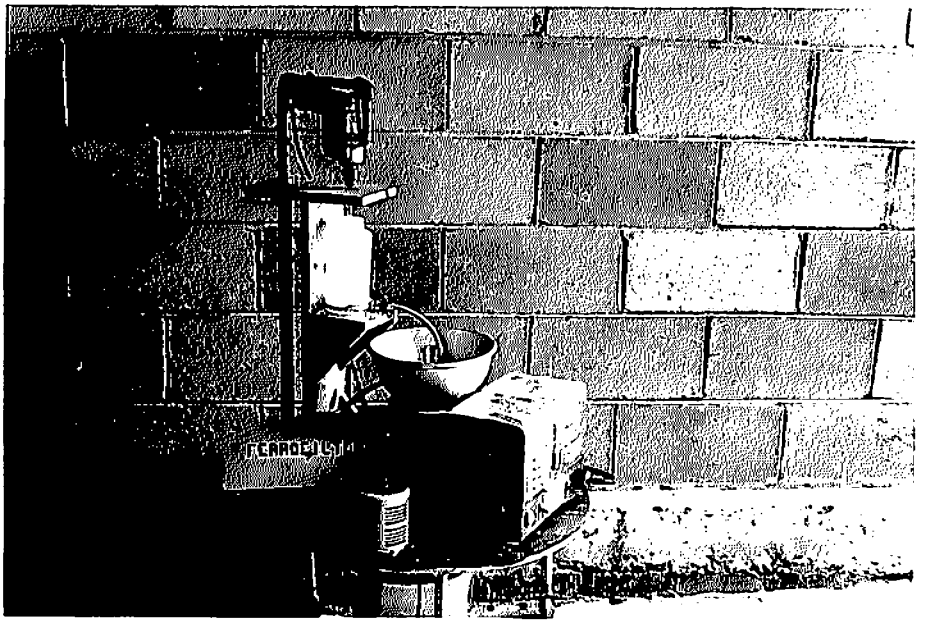


Flotation Beneficiation

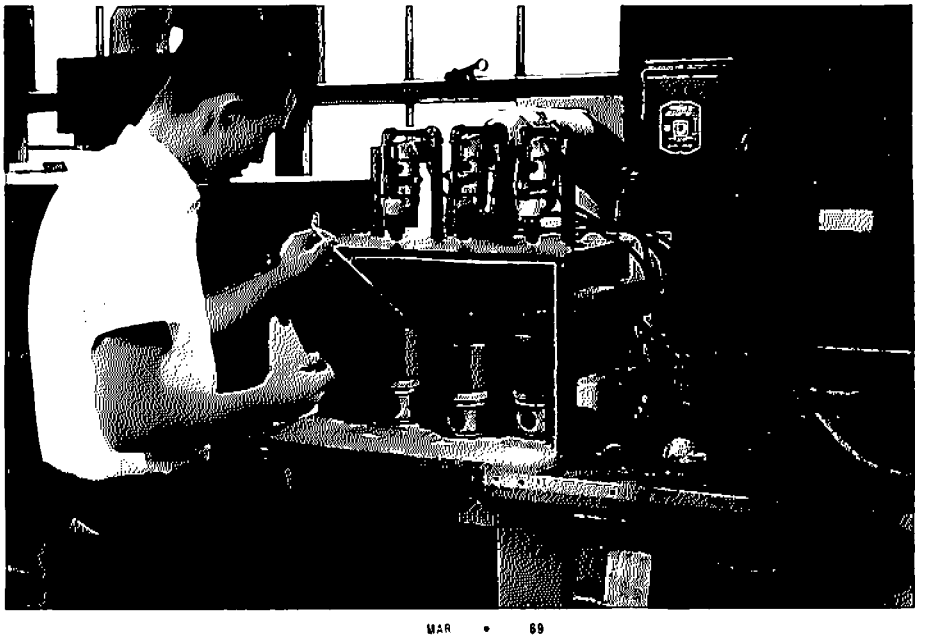


Mica Froth Product

Process Photographs



Conditioning and Wet Magnetic Separation



Acid Leaching