PILOT PLANT PROCESSING OF SILICA ORE COMPARING VARIOUS REAGENTS AND TECHNIQUES

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ABSTRACT

Extensive exploration is being undertaken in North Carolina and adjacent states to locate silica deposits from which high quality silica sand can be produced for the flat glass industry. Silica sand deposits are usually contaminated with various heavy minerals which are detrimental to flat glass making and therefore must be removed to make the silica useful. Specifications call for a product essentially not coarser than 40 mesh nor finer than 140 mesh. The maximum total iron is limited to 0.080% Fe_2O_3 with an acceptable variance of 0.040% maximum. Refractory heavy minerals (R.H.M.), such as zircon, kyanite, sillimanite, chromite, corundum, and andalusite, are limited to 0.200 grams of plus 70 mesh refractory minerals per 100 pounds of sand; this calculates to be 0.00044% by weight, 4.4 parts per million (ppm), or 1.0 pound per 138 tons.

Known flotation procedures were tried in bench-scale and pilot plant operations at the North Carolina State University Minerals Research Laboratory to remove contaminant minerals from silica ores. Some of these procedures were successful while others gave marginal products and were either too sensitive or too costly for efficient operation.

Because of the potential economic importance of producing glass-grade silica in North Carolina and the research-oriented

responsibilities of the Minerals Laboratory, a State-supported project was undertaken to develop new flotation procedures which might be efficient and economical for producing flat-glass-grade silica. This was accomplished in batch tests and verified through pilot plant operations. Silica products assaying less than 0.03% Fe_2O_3, and containing not more than 4.4 ppm of plus 70 mesh refractory minerals were produced by the new flotation procedure as well as with some of the other procedures investigated.

INTRODUCTION

Considerable interest has been shown by several companies in establishing mining operations in North Carolina and neighboring states for the production of flat-glass-grade silica products. This has been brought about by the construction of several plants for production of glass products, including one of the world's largest flat-glass plants - the L-O-F plant at Laurinburg, North Carolina.

Several companies solicited the services of the Minerals Research Laboratory for batch testing of sand samples, using established procedures or those of their choosing. Pilot plant operations were performed on ores from different areas in the State and from neighboring states. Some of these procedures were successful while others resulted in flowsheets which produced satisfactory glass-grade silica but which appeared to be excessively complicated. A State-supported

research project was undertaken to develop other flotation procedures for the efficient and economical removal of heavy minerals contaminants from silica ores. The ultimate objective of this research was the production of glass-grade silica from close proximity to North Carolina glass plants. An efficient and economical flotation procedure using an anionic detergent-type reagent was developed and found to be effective in batch tests. This reagent is of particular interest because it is biodegradable. Pilot plant tests produced products meeting Fe_2O_3 specifications but were marginal as to plus 70 mesh refractory minerals. Additional batch tests were conducted and the pilot plant modified. Ten pilot plant tests were then carried out comparing the new procedure with various other procedures, with emphasis placed on reagent types. Six of the ten pilot plant tests, including the new procedure, produced products meeting L-O-F specifications. This latest series of ten pilot plant tests is the principal subject of this report.

ORE

Approximately ten tons of ore from the Sand Hills area in the vicinity of Cognac, North Carolina was shipped by truck to the Laboratory for pilot plant processing. A representative sample of

the ore contained 0.51% heavy minerals in the deslimed plus 140 mesh fraction as determined by heavy liquid (sp gr 2.96) techniques. The average for the deslimed, plus 140 mesh feed to flotation for the ten pilot plant tests was 0.49% heavy minerals. The average chemical analyses of the deslimed floation feed for ten tests was: 0.17% Fe_2O_3, 0.39% Al_2O_3, 0.019% Na_2O, 0.032% K_2O, and 0.13% LOI. Approximately 44% of the ore was in the minus 40 plus 140 mesh size range, with 47.0% being coarser than 40 mesh and 9% being finer than 140 mesh.

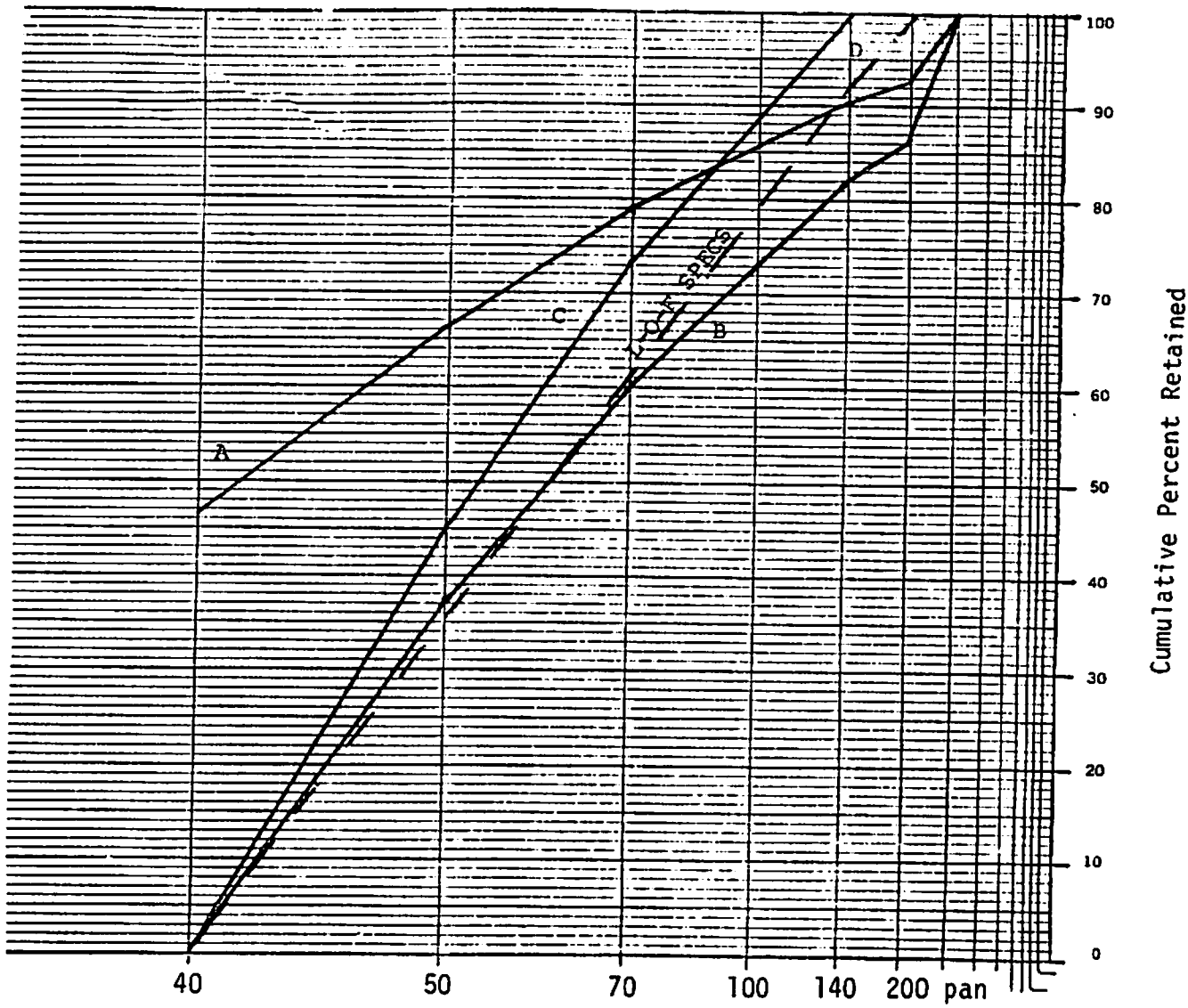
SIZING

Screen analyses of head feed ore, ore with plus 40 mesh fraction removed, and ore with plus 40 and minus 140 mesh fractions removed were compared with L-O-F size distribution specifications (see Figure 1). Removing the plus 40 mesh fraction would produce a product containing too much minus 140 mesh sands. The minus 140 mesh material could be removed in the desliming processing stage; however, this would leave a product coarser than that described in L-O-F size specifications. A pilot plant rod milling test was undertaken to adjust the grind so that deslimed ore could be rod milled to produce a product approximating the required size specifications (see Figure 2). Screen analyses of products from eight pilot plant tests are shown in Figures 4 and 5.

PILOT PLANT OPERATION

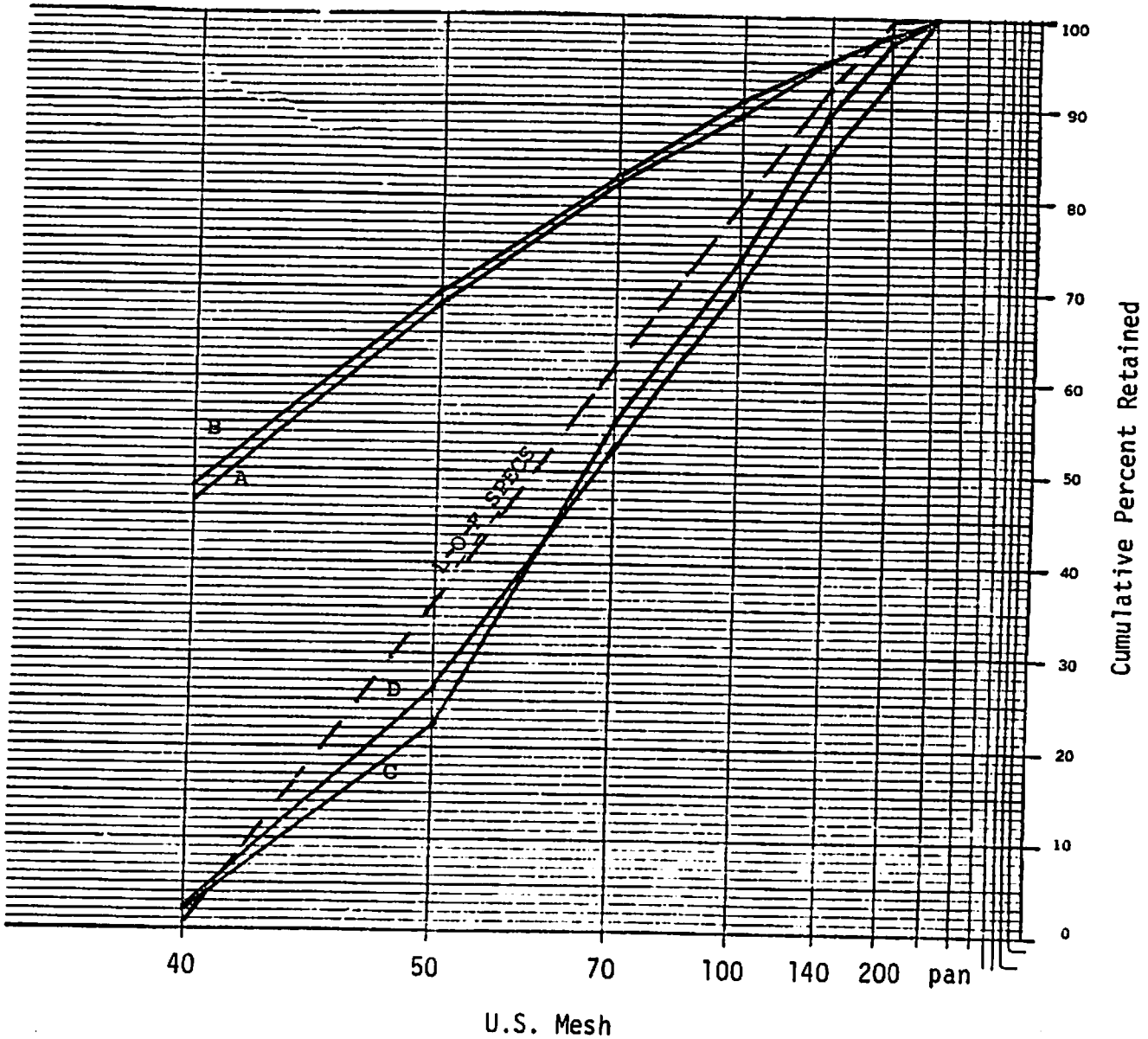
Ore was loaded into a hopper and fed with a belt conveyor at approximately 300 pounds per hour to a pulper where water was added and the pulp agitated for five minutes. The material was pumped to

Figure 1. Screen analyses - head feed, oversize removed, oversize and undersize removed,and L-O-F product specifications.



SCREEN SCALE RATIO 1.414				A Head Feed		B +40 Mesh Removed		C +40 & -140 Removed		D L-O-F Specs	
Openings Inches	Openings Milli-meters	Tyler Mesh	U. S. No.	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights
.185	4.699	4	4								
.131	3.327	6	6								
.093	2.362	8	8								
.065	1.651	10	12								
.046	1.168	14	16								0.00
.0328	.833	20	20								0.01
.0232	.589	28	30	25.0	25.0						
.0164	.417	35	40	21.7	46.7						0.10
.0116	.295	48	50	19.9	66.6	37.4	37.4	45.5	45.5		
.0082	.208	65	70	12.4	79.0	23.2	60.6	28.2	73.7		
.0058	.147	100	100	6.7	85.7	12.5	73.1	15.3	89.0		
.0041	.104	150	140	4.8	90.5	9.0	82.1	11.0	100.0		92.00
.0029	.074	200	200	2.3	92.8	4.2	86.3				99.50
			-200	7.2	100.0	13.7	100.0				
			Totals,	100.0		100.0		100.0			

Figure 2. Screen analyses - at various stages of pilot plant processing.



SCREEN SCALE RATIO 1.414				A Pulper Disc.		B Rod Mill Feed		C Rod Mill Disc.		D Silica Product	
Openings		Tyler Mesh	U. S. No.	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights
Inches	Millimeters										
.186	4.699	4	4								
.131	3.327	6	6								
.093	2.362	8	8								
.065	1.651	10	12								
.046	1.168	14	16								
.0328	.833	20	20								
.0232	.589	28	30								
.0164	.417	35	40	46.8	46.8	48.1	48.1	2.3	2.3	2.0	2.0
.0116	.295	48	50	21.7	68.5	21.8	69.9	24.3	26.6	20.7	22.7
.0082	.208	65	70	13.1	81.6	12.2	82.1	26.4	53.0	32.8	55.5
.0058	.147	100	100	7.7	89.3	8.8	90.9	17.6	70.6	18.0	73.5
.0041	.104	150	140	5.9	95.2	4.8	95.1	14.5	85.1	16.1	89.6
.0029	.074	200	200	3.0	98.2	2.8	98.5	8.1	93.2	8.1	97.7
			-200	1.8	100.0	1.5	100.0	6.8	100.0	2.3	100.0
			Totals,	100.0		100.0		100.0		100.0	

a cyclone. The cyclone overflow slimes went to waste, and the underflow gravitated to a rod mill. The ore was ground at 24% solids for a retention time of approximately 5 minutes. The mill discharge gravitated to a trommel screen, which was an integral part of the rod mill, for removal of plus 30 mesh oversize material. The screen undersize material was pumped to a cyclone for desliming. The cyclone overflow slimes went to waste, and the underflow gravitated to a spiral classifier for additional slime removal and dewatering preparatory to attrition scrubbing, in the tests where a scrubber was used (tests 8, 9, 10). In these tests, the high density material from the classifier was attrition scrubbed at 70 to 75% solids for 20 to 25 minutes in a pulp containing 2.0 pounds of 66° Baume H_2SO_4 per ton of ore (added as a 5% acid solution). The scrubber discharge material or the unscrubbed sand product from the first spiral classifier, depending on which pilot plant test was being conducted, was pumped to a cyclone for desliming. The cyclone overflow slimes went to waste,and the underflow material gravitated to a spiral classifier for additional slime removal and dewatering preparatory to conditioning. The discharge material from the classifier was conditioned for approximately 5 minutes at 60 to 65% solids in a pulp containing various reagents being investigated. The material gravitated to a second set of conditioners for an additional conditioning time of 6 minutes at 60 to 65% solids to insure sufficient coating of mineral particles. The conditioned material gravitated to the flotation cells where the contaminant minerals were removed as a froth product and the silica product was recovered in the machine underflow discharge.

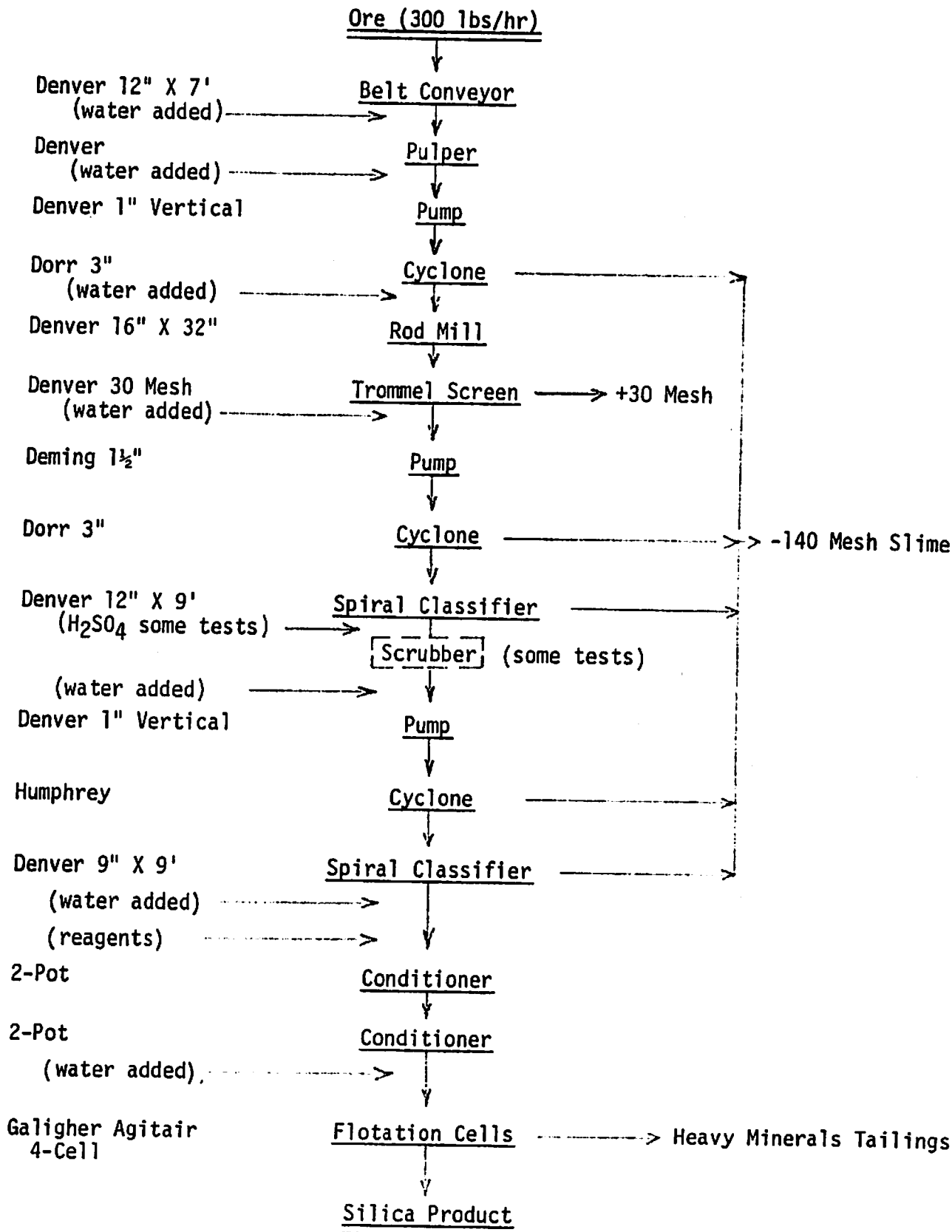


Figure 1. Pilot plant flowsheet

PROCESS CONTROL AND ANALYSES

Timed samples of ore feed, flotation feed, slimes, tailings, oversize, and silica product were taken during the testing to determine material balances. Chemical analyses were obtained on the feed to flotation, tailings, and silica product. Heavy liquid separation was used to determine heavy mineral content of feed to flotation, tailings, and silica product. Silica products were screened on 70 mesh, the plus 70 mesh and minus 70 mesh fractions were separated in heavy liquid, and heavy minerals content determined for the total product. Grain counts and mineral identifications were made on the plus 70 mesh sinks.* Size distributions of silica products were obtained by screening on a Ro-Tap. Water distribution to equipment was monitored with flowrators and water consumed determined with a totalizing meter.

NEW PROCEDURE

Four of the ten pilot plant runs used the new heavy mineral flotation process which was developed at the Minerals Research Laboratory. The primary feature of this procedure is the use of detergent-type reagents, such as sodium alcohol ether sulfate (Tex-Wet 1158) and a dodecylbenzene sulfonic acid (Tex-Wet 1197), as heavy minerals collectors in an acid circuit for froth flotation. These reagents were obtained from Intex Products, Inc., Greenville, South Carolina; however, other brands of like chemicals were found to be satisfactory in batch tests. The sodium alcohol ether sulfate-dodecylbenzene

* Grain counts and minerals identification were performed by Mr. Carl Merschat, geologist with the Div. of Resource Planning and Evaluation.

sulfonic acid collector adheres to the heavy minerals rendering them hydrophobic, thereby allowing attachment of air bubbles and removal of the heavy minerals as a froth product. Silica is removed along the bottom of the chamber, not being affected by the collector and being suppressed by the acid media at low pH.

OTHER PROCEDURES

Pilot plant tests were also performed using fatty acid and petroleum sulfonate collectors, and combinations of these collectors with the experimental detergent-type reagent described above. Tests were run both without (tests 1 through 7) and with (tests 8, 9, 10) scrubbing.

RESULTS

The first two pilot plant runs involved equipment adjustment and pilot plant tune-up. Tex-Wet reagent was used in both tests, with the lower amount of reagent giving the better grade product. These tests did not meet plus 70 mesh refractory minerals specifications. Test 3, using fatty acid with the Tex-Wet reagent, met specs. Test 4 produced the best product of all tests and involved the use of petroleum sulfonate as collector. Test 5, using Tex-Wet reagent alone, met specs. Test 6, using reduced amount of petroleum sulfonate along with Tex-Wet reagent, met specs. Test 7 involved a process similar to that of a commercial sand company, using sodium hydroxide, fatty acid, fuel oil, and pine oil, but it did not meet specs. Test 8, using scrubbing and Tex-Wet reagent, was borderline

on specifications (0.22 vs 0.20 grams plus 70 refractory minerals per 100 pounds sand). Test 9, using scrubbing and fatty acid along with Tex-Wet reagent, met specs. Test 10, using scrubbing and reduced amount of petroleum sulfonate and Tex-Wet reagent, did not meet specifications. Eighty to eighty-five percent of the ore was recovered as a silica product in seven of the tests. The lowest (62.9%) recovery was in test 7, using sodium hydroxide, fatty acid, fuel oil, and pine oil. Test 4, using petroleum sulfonate, gave a recovery of 74.8%, and test 8, using scrubbing and Tex-Wet reagent, gave a recovery of 77.0%.

Data pertaining to tests are included in Tables I through X.

CONCLUSIONS

Five of the ten pilot plant tests met specifications for glass-grade sand, and one test was borderline. The Tex-Wet reagent was found to be effective as a collector for heavy minerals contaminants by itself and in combination with other reagents. The Tex-Wet reagent produces a voluminous froth which appears excessive; however, the froth dissipates rapidly as observed at a commercial plant where it was field tested. A lack of a good froth was experienced when using petroleum sulfonate or fatty acid (Pamak-25) float. A good froth was obtained by using Tex-Wet in combination with either of the two aforementioned reagents. Despite the lack of a good froth using petroleum sulfonate, this float produced the best silica product. Tex-Wet reagent is biodegradable which gives it a good selling point

when it comes to stream pollution problems.

All of the tests were single floats, and scrubbing was not required to produce a silica product meeting refractory mineral specifications.

The tests provided some valuable data for companies desiring to produce high-grade silica products. With the ever-changing availability of reagents, particularly the petrochemicals, and the fluctuating prices, it is good to have several alternatives for contaminant minerals removal.

PILOT PLANT TESTS

Test No. 1

This test involved use of crowder plates in float cells to assist in froth removal. No scrubbing was used. One pound of 66° Baume H_2SO_4 per ton of ore (added as a 5% acid solution) was added to the first conditioner. Two pounds of Tex-Wet reagent* per ton of ore were added to the second set of conditioners.

The product contained an excessive amount of plus 70 mesh refractory minerals. Detailed data for Test 1 are shown on Table I.

Test No. 2

This test was similar to Test 1 except collector was reduced to 1.0 pound per ton of ore (and added to second set of conditioners instead of first conditioners). The silica product was better than

*This reagent consisted of a 5% solution of equally proportioned reagents obtained from Intex Products, Inc., Greenville, S. C.: TW-1197, sodium alcohol ether sulfate; and TW-1158, dodecylbenzene sulfonic acid.

TABLE I

PILOT PLANT TEST NO. 1

Sample	Physical Data: Wt %	% H.M.	+70 Mesh RHM*: Gr/100 lbs	+70 Mesh RHM*: ppm	Chemical Analyses, %: Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	0.5								
H.M. Float	1.3	29.30			5.05	0.36	-	-	-
-140 M. Slime	13.4								
Silica Prod.	84.8	0.076	5.46	120	0.082	0.025	0.009	0.009	0.15
Total	100.0	0.460							
Flot. Feed (-30+140 M.)	86.1	0.510			0.203	0.720	0.020	0.040	0.10
Head Feed	100.0	0.460							

Process	Conditions: Time (Min)	% Solids	pH	Reagents (lbs/ton of feed): H_2SO_4	TW-1197	TW-1158
Belt Feeder	-	95				
Pulper	4.2	40				
#1 Pump	-	9				
#1 Cyclone U'flow	-	39				
Rod Mill	4.9	29				
Trommel Screen	-	-				
#2 Pump	-	4				
#2 Cyclone U'flow	-	17				
#1 Spiral Classifier	6.1	70				
#3 Pump	-	9				
#3 Cyclone U'flow	-	13				
#2 Spiral Classifier	6.2	70				
#1 Conditioner	5.1	60	1.99	1.0		-
#2 Conditioner	6.3	60	1.96			2.0
Float Cells	2.4	13	2.90	-		-

Remarks:

Feed Rate - 300 pounds per hour (dry basis).
Water Used - 12,790 gallons per ton of ore.

* R.H.M. = Refractory heavy minerals.

TABLE II

PILOT PLANT TEST NO. 2

Sample	Physical Data				Chemical Analyses, %				
	Wt %	% H.M.	+70 Mesh RHM Gr/100 lbs	ppm	Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	0.5								
H.M. Float	1.8	24.16			3.85	0.29	-	-	-
-140 M. Slime	14.2								
Silica Prod.	83.5	0.044	1.35	30	0.036	0.005	0.005	0.005	0.08
Total	100.0	0.472							
Flot. Feed (-30+140 M.)	85.3	0.50			0.171	0.120	0.020	0.030	0.10
Head Feed	100.0	0.472							

Process	Conditions			Reagents (lbs/ton of feed)	
	Time (Min)	% Solids	pH	H_2SO_4	TW-1197 TW-1158
Belt Feeder	-	95			
Pulper	4.9	42			
#1 Pump	-	7			
#1 Cyclone U'flow	-	40			
Rod Mill	3.3	29			
Trommel Screen	-	-			
#2 Pump	-	4			
#2 Cyclone U'flow	-	12			
#1 Spiral Classifier	6.4	70			
#3 Pump	-	18			
#3 Cyclone U'flow	-	18			
#2 Spiral Classifier	6.5	70			
#1 Conditioner	5.7	58	1.97	1.0	-
#2 Conditioner	7.1	58	2.10	-	1.0
Float Cells	2.1	11	2.26	-	-

Remarks:

Feed Rate - 286 pounds per hour (dry basis).
Water Used - 15,027 gallons per ton of ore.
Crowder plates used in float cells to assist in froth removal.

previous test but still contained too much (30 ppm) plus 70 mesh refractory minerals. Crowder plates appeared to be detrimental, possibly causing bubbles to burst prematurely and release entrapped heavy minerals contaminants. The plates were removed before Test 3. Test 2 data are shown on Table II.

Test No. 3

One-half pound of fatty acid* and one-half pound of Tex-Wet per ton of ore were added to second set of conditioners. H_2SO_4 was added to first conditioners. Crowder plates had been removed before this test. The silica product contained 0.13 grams of plus 70 mesh refractory minerals per 100 pounds of sand. Test 3 data are shown on Table III.

Test No. 4

Two and one-half pounds of H_2SO_4 per ton of ore was added to first conditioners. One pound of petroleum sulfonate** and 0.10 pound of frother*** per ton of ore were added to the second conditioners. The silica product contained 0.003 grams of plus 70 mesh refractory minerals per 100 pounds of sand. Test 4 data are shown on Table IV.

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*Pamak-25 obtained from Hercules Incorporated, Wilmington, Delaware.

**M-70 obtained from Hunt Chemicals, Marion, North Carolina.

***F-75 glycol frother obtained from American Cyanamid Company, Wayne, New Jersey.

TABLE III

PILOT PLANT TEST NO. 3

	Physical Data				Chemical Analyses, %				
Sample	Wt %	% H.M.	+70 Mesh RHM Gr/100 lbs	ppm	Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	0.2								
H.M. Float	2.3	23.50			4.30	1.10	-	-	-
-140 M. Slime	13.2								
Silica Prod.	84.3	0.0095	0.13	2.9	0.024	0.005	0.008	0.012	0.05
Total	100.0	0.55							
Flot. Feed (-30+140 M.)	86.5	0.56			0.170	0.460	0.020	0.040	0.05
Head Feed	100.0	0.55							

	Conditions			Reagents (lbs/ton of feed)		
Process	Time (Min)	% Solids	pH	H_2SO_4	TW-1197 TW-1158	Pamak-25
Belt Feeder	-	95				
Pulper	3.9	39				
#1 Pump	-	7				
#1 Cyclone U'flow	-	32				
Rod Mill	2	20				
Trommel Screen	-	-				
#2 Pump	-	3				
#2 Cyclone U'flow	-	12				
#1 Spiral Classifier	6.0	70				
#3 Pump	-	19				
#3 Cyclone U'flow	-	19				
#2 Spiral Classifier	6.0	70				
#1 Conditioner	5.1	58	2.04	1.0	-	-
#2 Conditioner	6.4	58	2.25	-	0.5	0.5
Float Cells	2.2	12	2.88	-	-	-

Remarks:

Feed Rate - 322 pounds per hour (dry basis)
Water Used - 13,405 gallons per ton of ore.
Crowder plates removed from float cells for this and all subsequent tests.

TABLE IV

PILOT PLANT TEST NO. 4

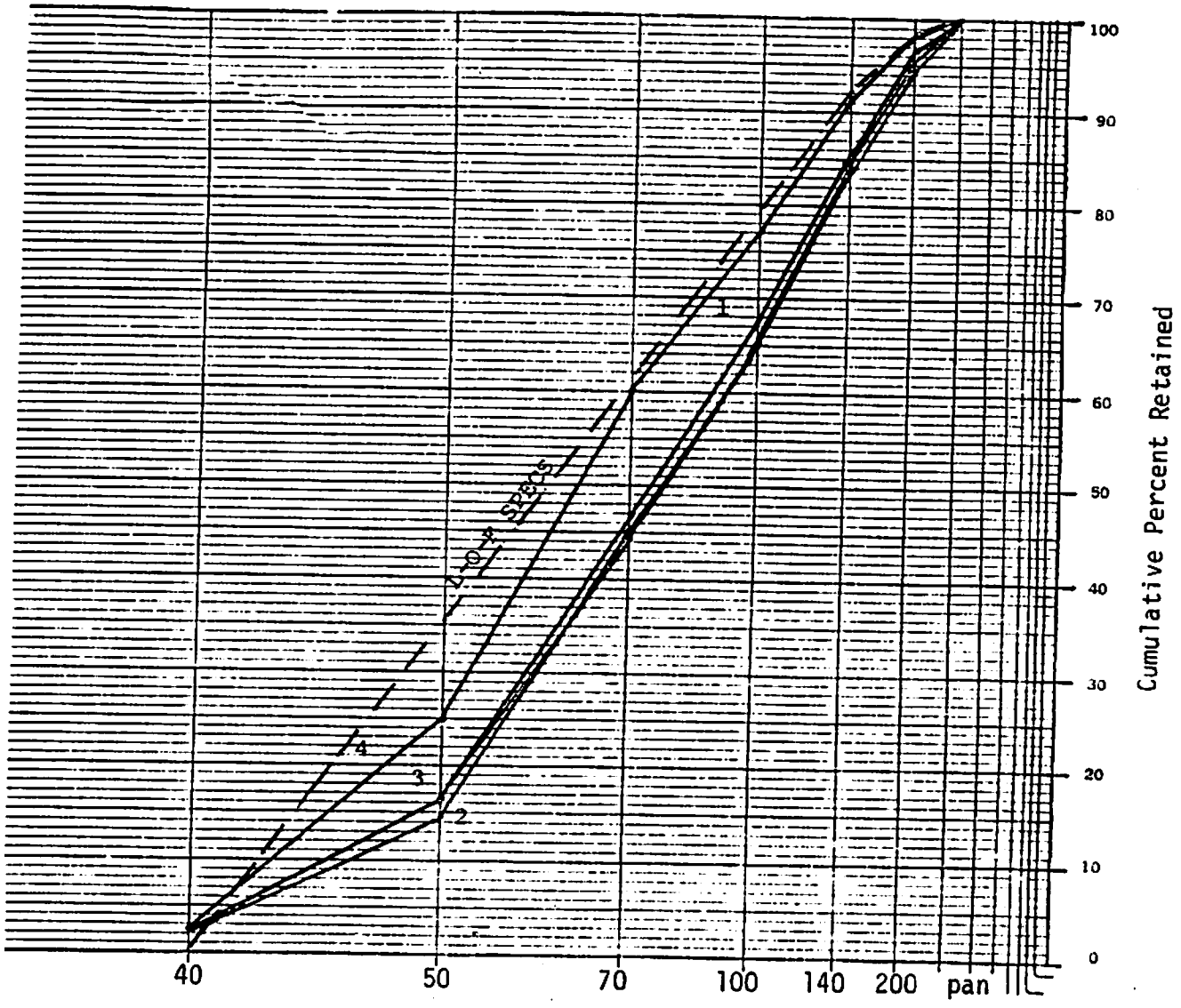
Sample	Physical Data: Wt %	% H.M.	+70 Mesh RHM Gr/100 lbs	ppm	Chemical Analyses, %: Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	2.0								
H.M. Float	9.3	5.587			0.88	0.84	-	-	-
-140 M. Slime	13.9								
Silica Prod.	74.8	0.0053	0.003	0.07	0.017	0.06	0.007	0.006	0.04
Total	100.0	0.52							
Flot. Feed (-30+140 M.)	84.1	0.52			0.140	0.11	0.02	0.02	0.09
Head Feed	100.0								

Process	Conditions: Time (Min)	% Solids	pH	Reagents (lbs/ton of feed): H_2SO_4	M-70	F-75
Belt Feeder	-	95				
Pulper	1.7	20				
#1 Pump	-	7				
#1 Cyclone U'flow	-	38				
Rod Mill	2.3	21				
Trommel Screen	-	-				
#2 Pump	-	3				
#2 Cyclone U'flow	-	14				
#1 Spiral Classifier	5.9	70				
#3 Pump	-	17				
#3 Cyclone U'flow	-	17				
#2 Spiral Classifier	5.9	70				
#1 Conditioner	6.3	61	1.77	2.5	-	-
#2 Conditioner	7.9	61	1.95	-	1.0	0.1
Float Cells	2.5	11	2.66	-	-	-

Remarks:

Feed Rate - 314 pounds per hour (dry basis)
Water Used - 12,983 gallons per ton of ore.

Figure 4. Screen analyses - pilot plant silica product.



Pilot Plant Test

SCREEN SCALE RATIO 1.414				#1		#2		#3		#4	
Openings		Tyler Mesh	U. S. No.	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights
Inches	Millimeters										
.185	4.699	4	4								
.131	3.327	6	6								
.093	2.362	8	8								
.065	1.651	10	12								
.046	1.168	14	16								
.0328	.833	20	20								
.0232	.589	28	30								
.0164	.417	35	40	2.0	2.0	2.0	2.0	2.5	2.5	2.5	2.5
.0116	.295	48	50	14.3	16.3	12.5	14.5	13.7	16.2	22.7	25.2
.0082	.208	65	70	30.6	46.9	30.9	45.4	28.2	44.4	34.9	60.1
.0058	.147	100	100	20.7	67.6	19.7	65.1	20.8	65.2	17.0	77.1
.0041	.104	150	140	18.3	85.9	18.9	84.0	19.6	84.8	14.8	91.9
.0029	.074	200	200	10.7	96.6	10.5	94.5	10.6	95.4	6.6	98.5
			-200	3.5	100.0	5.5	100.0	4.6	100.0	1.5	100.0
			Totals	100.0		100.0		100.0			

Test No. 5

One pound of H_2SO_4 and one pound of Tex-Wet 1158-1197 (see footnote Test 1) per ton of ore was added to first conditioner. The silica product contained 0.18 grams of plus 70 mesh refractory minerals per 100 pounds of sand. Test 5 data are shown on Table V.

Test No. 6

Two and one-half pounds of H_2SO_4 per ton of ore was added to first conditioner. One-half pound of petroleum sulfonate (M-70) and one-half pound of Tex-Wet 1158-1197 per ton of ore were added to second conditioners. The silica product contained 0.08 grams of plus 70 mesh refractory minerals per 100 pounds of sand. Detailed data for Test 6 are shown on Table VI.

Test No. 7

This test was intended to duplicate a processing technique used in a commercial sand plant in Tennessee. A satisfactory separation could not be made, and the float was difficult to control. The silica product contained an excessive amount of plus 70 mesh refractory minerals. Test 7 data are shown on Table VII.

Test No. 8

An attrition scrubber was used in this test for additional cleaning of mineral grains for reagent attachment. Two pounds of H_2SO_4 per ton of ore was fed to the scrubber. One pound of H_2SO_4 and one pound of Tex-Wet 1158-1197 were added to the first conditioner. The silica product contained 0.22 grams of plus 70 mesh refractory minerals per 100 pounds of sand. Detailed data for Test 8 are shown on Table VIII.

TABLE V

PILOT PLANT TEST NO. 5

Sample	Physical Data: Wt %	% H.M.	+70 Mesh RHM Gr/100 lbs	+70 Mesh RHM ppm	Chemical Analyses, %: Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	3.8								
H.M. Float	1.5	22.76			4.10	1.23	-	-	-
-140 M. Slime	13.4								
Silica Prod.	81.3	0.0092	0.18	3.9	0.022	0.06	0.006	0.010	0.06
Total	100.0	0.349							
Flot. Feed (-30+140 M.)	82.8	0.48			0.15	0.460	0.015	0.025	0.09
Head Feed	100.0	0.349							

Process	Conditions: Time (Min)	% Solids	pH	Reagents (lbs/ton of feed): H_2SO_4	TW-1197 TW-1158
Belt Feeder	-	95			
Pulper	4.4	38			
#1 Pump	-	13			
#1 Cyclone U'flow	-	40			
Rod Mill	2.4	24			
Trommel Screen	-	-			
#2 Pump	-	4			
#2 Cyclone U'flow	-	20			
#1 Spiral Classifier	5.5	70			
#3 Pump	-	19			
#3 Cyclone U'flow	-	19			
#2 Spiral Classifier	5.5	70			
#1 Conditioner	5.4	60	1.8	1.0	1.0
#2 Conditioner	6.7	60	1.9	-	-
Float Cells	2.2	12	2.7	-	-

Remarks:

Feed Rate - 338 pounds per hour (dry basis)
Water Used - 11,877 gallons per ton of ore.
Collector reagent added to first pot in #1 conditioner

TABLE VI

PILOT PLANT TEST NO. 6

Sample	Physical Data: Wt %	% H.M.	+70 Mesh RHM Gr/100 lbs	+70 Mesh RHM ppm	Chemical Analyses, %: Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	1.3								
H.M. Float	2.1	16.173			3.00	1.00	-	-	-
-140 M. Slime	13.4								
Silica Prod.	83.2	0.0122	0.08	1.76	0.023	0.07	0.005	0.007	0.11
Total	100.0	0.350							
Flot. Feed (-30+140 M.)	85.3	0.43			0.16	0.50	0.02	0.04	0.24
Head Feed	100.0	0.35							

Process	Conditions: Time (Min)	% Solids	pH	Reagents (lbs/ton of feed): H_2SO_4	M-70	TW-1197 TW-1158
Belt Feeder	-	95				
Pulper	2.7	35				
#1 Pump	-	9				
#1 Cyclone U'flow	-	37				
Rod Mill	1.9	23				
Trommel Screen	-	-				
#2 Pump	-	4				
#2 Cyclone U'flow	-	22				
#1 Spiral Classifier	5.4	70				
#3 Pump	-	13				
#3 Cyclone U'flow	-	13				
#2 Spiral Classifier	5.4	70				
#1 Conditioner	5.5	63	1.75	2.5	-	-
#2 Conditioner	6.9	63	2.04	-	0.5	0.5
Float Cells	2.3	13	2.47	-	-	-

Remarks:

Feed Rate - 345 pounds per hour (dry basis)
Water Used - 13,139 gallons per ton of ore.

TABLE VII

PILOT PLANT TEST NO. 7

	Physical Data				Chemical Analyses, %				
			+70 Mesh RHM						
Sample	Wt %	% H.M.	Gr/100 lbs	ppm	Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	2.3								
H.M. Float	1.4	3.70			0.97	0.52	-	-	-
-140 M. Slime	33.4								
Silica Prod.	62.9	0.34	(excessive)		0.094	0.06	0.006	0.017	0.10
Total	100.0								
Flot. Feed (-30+140 M.)	64.3	0.58			0.21	0.35	0.024	0.040	0.19
Head Feed	100.0	0.27							

	Conditions			Reagents (lbs/ton of feed)			
Process	Time (Min)	% Solids	pH	NaOH	Pamak-25	Fuel Oil	Pine Oil
Belt Feeder	-	95					
Pumper	3.7	38					
#1 Pump	-	9					
#1 Cyclone U'flow	-	38					
Rod Mill	2.4	20					
Trommel Screen	-	-					
#2 Pump	-	3					
#2 Cyclone U'flow	-	20					
#1 Spiral Class.	5.1	70					
#3 Pump	-	14					
#3 Cycl. U'flow	-	14					
#2 Spiral Class.	6.7	70					
#1 Conditioner	8.0	65	6.50	0.3	1.0	0.5	0.1
#2 Conditioner	10.0	65	7.60				
Float Cells	2.5	10	8.33				

Remarks:

Feed Rate - 326 pounds per hour (dry basis)
Water Used - 13,656 gallons per ton of ore

TABLE VIII

PILOT PLANT TEST NO. 8

Sample	Physical Data: Wt %	% H.M.	+70 Mesh RHM Gr/100 lbs	+70 Mesh RHM ppm	Chemical Analyses, %: Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	3.6								
H.M. Float	6.3	5.30			0.88	0.398	-	-	-
-140 M. Slime	13.1								
Silica Prod.	77.0	0.0167	0.22	4.8	0.027	0.044	0.0062	0.0061	0.10
Total	100.0								
Flot. Feed (-30+140 M.)	83.3	0.369			0.114	0.41	0.0184	0.0312	0.12
Head Feed	100.0	0.357							

Process	Conditions: Time (Min)	% Solids	pH	Reagents (lbs/ton of feed): H_2SO_4	TW-1197 TW-1158
Belt Feeder	-	95			
Pulper	3.6	38			
#1 Pump	-	9			
#1 Cyclone U'flow	-	35			
Rod Mill	2.1	22			
Trommel Screen	-	-			
#2 Pump	-	4			
#2 Cyclone U'flow	-	25			
#1 Spiral Classifier	5.6	70			
Scrubber	22.0	71		2.0	-
#3 Pump	-	22			
#3 Cyclone U'flow	-	22			
#2 Spiral Classifier	5.6	70			
#1 Conditioner	5.0	58	2.42	1.0	1.0
#2 Conditioner	6.2	58	2.45		
Float Cells	2.7	15	3.11		

Remarks:

Feed Rate - 335 pounds per hour (dry basis)
Water Used - 11,111 gallons per ton of ore

Test No. 9

An attrition scrubber was used in this test for additional cleaning of mineral grains for reagent attachment. Two pounds of H_2SO_4 per ton of ore was fed to the scrubber. One pound of H_2SO_4 0.4 pound of fatty acid (Pamak-25), and 0.2 pound of Tex-Wet 1158-1197 per ton of ore were added to the first conditioner. The silica product contained 0.20 grams of plus 70 mesh refractory minerals per 100 pounds of sand. Detailed data for Test 9 are shown on Table IX.

Test No. 10

An attrition scrubber was used for this test for additional cleaning of mineral grains for reagent attachment. A considerably reduced reagent charge was employed for this run. Two pounds of H_2SO_4 per ton of ore was fed to the scrubber. One pound of H_2SO_4, 0.2 pounds of petroleum sulfonate (M-70), and 0.2 pound of Tex-Wet 1158-1197 were added to first conditioner. The silica product contained an excessive amount of plus 70 mesh refractory minerals. Test 10 data are shown on Table X.

TABLE IX

PILOT PLANT TEST NO. 9

	Physical Data				Chemical Analyses, %				
			+70 Mesh RHM						
Sample	Wt %	% H.M.	Gr/100 lbs	ppm	Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	2.4								
H.M. Float	2.0	18.75			3.50	0.80	-	-	-
-140 M. Slime	11.1								
Silica Prod.	84.5	0.0270	0.20	4.4	0.0270	0.039	0.0054	0.0034	0.04
Total	100.0								
Flot. Feed (-30+140 M.)	86.5	0.440			0.17	0.398	0.0140	0.0184	0.15
Head Feed	100.0	0.398							

	Conditions			Reagents (lbs/ton of feed)		
Process	Time (Min)	% Solids	pH	H_2SO_4	Pamak-25	TW-1197 TW-1158
Belt Feeder	-	95				
Pulper	3.7	35				
#1 Pump	-	22				
#1 Cyclone U'flow	-	41				
Rod Mill	2.4	23				
Trommel Screen	-	-				
#2 Pump	-	4				
#2 Cyclone U'flow	-	25				
#1 Spiral Classifier	6.3	70				
Scrubber	25.0	72		2.0		
#3 Pump	-	17				
#3 Cyclone U'flow	-	17				
#2 Spiral Classifier	6.3	70				
#1 Conditioner	7.1	67	1.82	1.0	0.4	0.2
#2 Conditioner	8.9	67	1.82			
Float Cells	2.1	11	2.70			

Remarks:

Feed Rate - 294 pounds per hour (dry basis)
Water Used - 12,932 gallons per ton of ore

TABLE X

PILOT PLANT TEST NO. 10

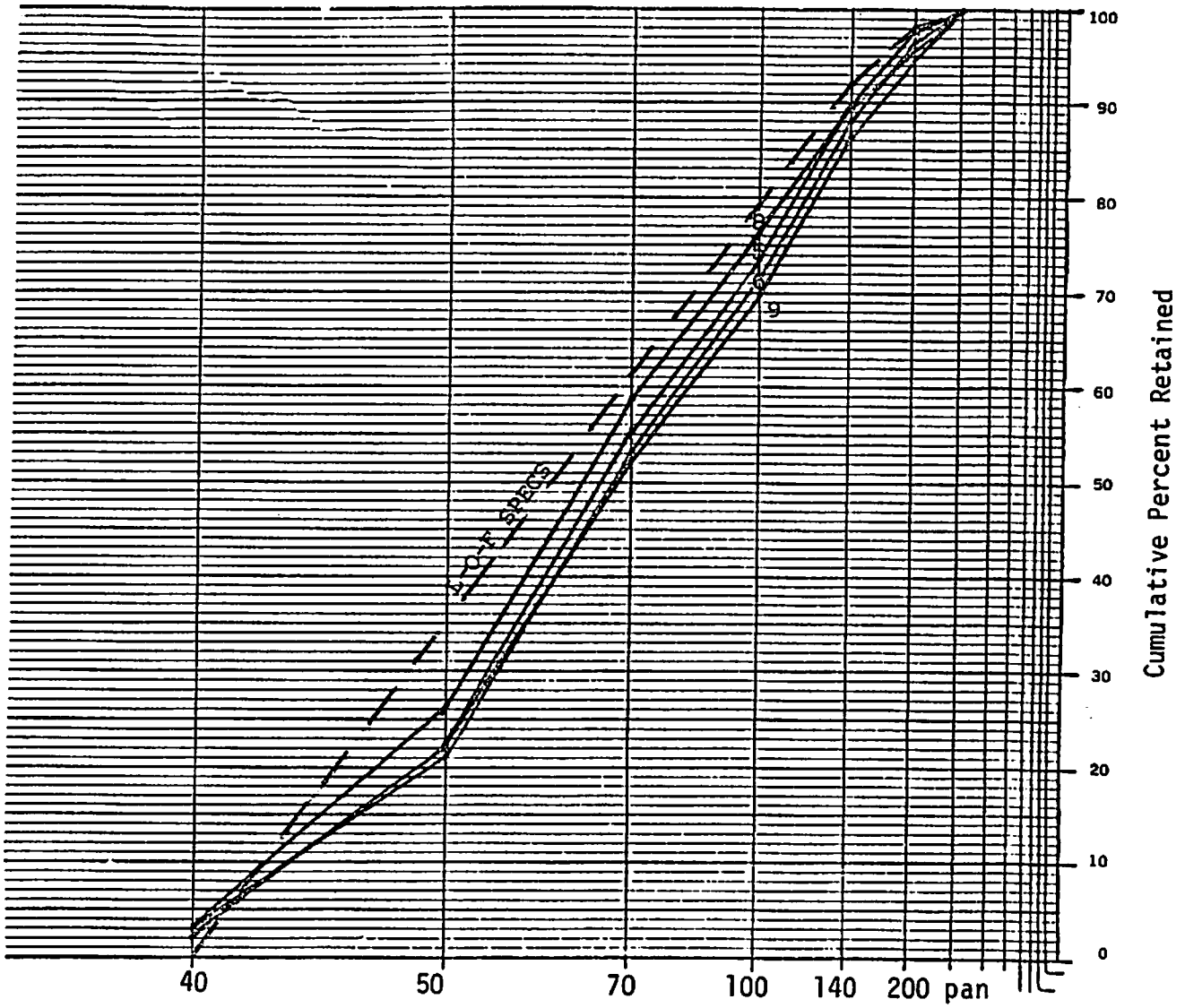
	Physical Data				Chemical Analyses, %				
			+70 Mesh RHM						
Sample	Wt %	% H.M.	Gr/100 lbs	ppm	Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	0.9								
H.M. Float	0.9	17.17			3.2	1.2	-	-	-
-140 M. Slime	11.3								
Silica Prod.	86.9	0.23	(excessive)		0.08	0.07	0.004	0.007	0.10
Total	100.0								
Flot. Feed (-30+140 M.)	87.8	0.471			0.18	0.65	0.004	0.007	0.10
Head Feed	100.0	0.354							

	Conditions			Reagents (lbs/ton of feed)		
Process	Time (Min)	% Solids	pH	H_2SO_4	M-70	TW-1197 TW-1158
Belt Feeder	-	95				
Pulper	4.1	35				
#1 Pump	-	8				
#1 Cyclone U'flow	-	27				
Rod Mill	1.9	17				
Trommel Screen	-	-				
#2 Pump	-	6				
#2 Cyclone U'flow	-	22				
#1 Spiral Classifier	7.0	70				
Scrubber	25.0	70		2.0		
#3 Pump	-	10				
#3 Cyclone U'flow	-	10				
#2 Spiral Classifier	7.0	70				
#1 Conditioner	6.4	61	1.53	1.0	0.2	0.2
#2 Conditioner	8.0	61	1.90			
Float Cells	2.6	11	2.59			

Remarks:

Feed Rate - 265 pounds per hour (dry basis)
Water Used - 17,447 gallons per ton of ore

Figure 5. Screen analyses - pilot plant silica product.



Pilot Plant Test

SCREEN SCALE RATIO 1.414				#5		#6		#8		#9	
Openings		Tyler Mesh	U. S. No.	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights
Inches	Milli-meters										
.185	4.699	4	4								
.131	3.327	6	6								
.093	2.362	8	8								
.065	1.651	10	12								
.046	1.168	14	16								
.0328	.833	20	20								
.0232	.589	28	30								
.0164	.417	35	40	2.0	2.0	3.0	3.0	3.2	3.2	2.9	2.9
.0116	.295	48	60	20.7	22.7	18.4	21.4	23.2	26.4	19.1	22.0
.0082	.208	65	70	32.8	55.5	31.8	53.2	32.4	58.8	30.3	52.3
.0058	.147	100	100	18.0	73.5	18.2	71.4	17.3	76.1	17.5	69.8
.0041	.104	150	140	16.1	89.6	16.6	88.0	13.5	89.6	16.4	86.2
.0029	.074	200	200	8.1	97.7	8.6	96.6	6.3	95.9	8.8	95.0
			-200	2.3	100.0	3.4	100.0	4.1	100.0	5.0	100.0
			Totals	100.0		100.0					