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PILOT PLANT EVALUATION OF AN ANIONIC DETERGENT-TYPE REAGENT FOR BENEFICIATION OF A GLASS SAND

by
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ABSTRACT

Extensive exploration is being undertaken in North Carolina to locate 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 must be removed to make the silica useful for flat-glass production.

In previous work at the North Carolina State University's Minerals Research Laboratory, known flotation procedures were evaluated in bench-scale and pilot plant operations for removing contaminant minerals from silica ores. A procedure involving the use of petroleum sulfonate as a flotation reagent was satisfactory, while other procedures gave marginal results or were too complicated to be economically feasible.

Because of the potential economic importance of producing glass-grade silica in North Carolina, additional research was undertaken to develop a better procedure for producing flat-glass-grade silica. Batch tests revealed that anionic detergent-type reagents had considerable promise. Pilot plant tests were run to compare the effectiveness of anionic detergent-type reagents with petroleum sulfonate in the removal of heavy minerals from a potential glass sand. Another slightly modified commercial procedure also was evaluated for comparison purposes. In the pilot plant trials, utilization of the anionic detergent flotation reagents did not produce quite as good results as petroleum sulfonate. It is likely that flat-glass-grade sand could be produced with the anionic detergent reagents if more extensive pilot plant tests were conducted. This may become desirable in the future if it becomes necessary to utilize flotation reagents which are rapidly biodegradable.

INTRODUCTION

Considerable interest has been shown by several companies in establishing mining operations in North Carolina and neighboring states for the production of silica for use in making glass. This interest has been intensified by the construction of several plants for production of glass products, including one of the world's largest flat-glass plants -- the Libbey-Owens-Ford Company (L-O-F) plant at Laurinburg, North Carolina.

In the past, several companies solicited the services of the Minerals Research Laboratory for batch testing of sand samples using established procedures or experimental procedures of their choosing. Pilot-plant operations were performed on ores from different parts of North Carolina and neighboring states. Some procedures were successful; others produced satisfactory glass-grade silica but had excessively complicated flowsheets.

State-supported research 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 sites near existing 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 rapidly biodegradable. A pilot plant was constructed to test the procedure in a continuous operation. Process variables were standardized in a series of preliminary runs so that direct comparisons could be made of the effects of various types and combinations of reagents. It was realized that this would not permit optimum adjustment or product grade for each test. Ten pilot-plant tests were carried out comparing the new procedure with a commercial procedure and a slightly modified commercial procedure. Six tests, including the new procedure, produced products containing from 0.041 to 0.527 grams of plus-70 mesh refractory heavy minerals per 100 pounds of sand. More than 98.6% of the heavy minerals contaminants were removed in these tests.

PROCEDURE

Ore

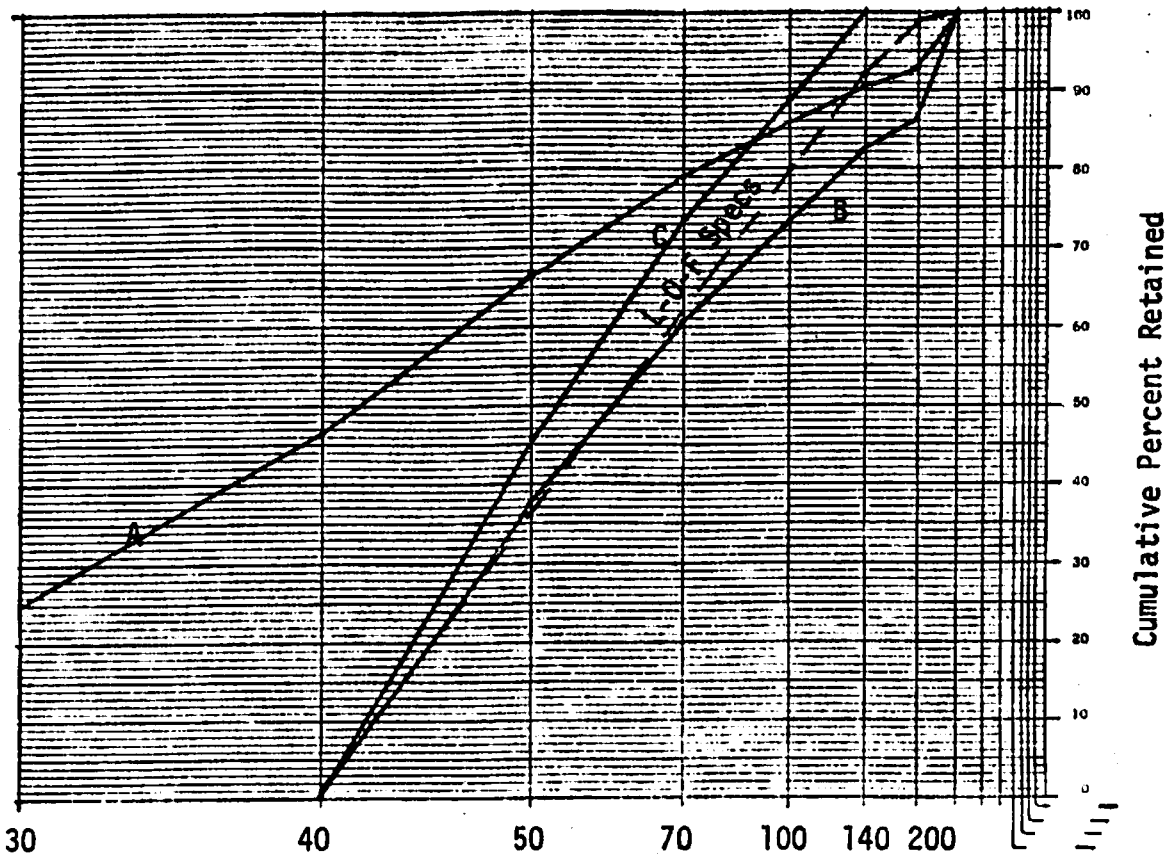
Approximately ten tons of ore from the Sand Hills area in the vicinity of Cognac, North Carolina, were shipped by truck to the Minerals Research Laboratory for pilot plant processing. A representative sample of the ore contained 0.51% heavy minerals in the deslimed plus 140 mesh fraction as determined 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 analysis of the deslimed flotation 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 (loss on ignition).

Specifications

The L-O-F glass sand specifications used in this research were: 0.080% Fe_2O_3 content with an acceptable variance of 0.040% maximum and 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 in 100 pounds of sand. The latter is equivalent to 0.00044% by weight, 4.4 ppm, or 1.0 pound in 138 tons. In addition, size specifications are included as discussed below. L-O-F glass sand specifications were used for two reasons. First, these specifications are typical of those in the flat-glass industry, and they are considerably more stringent than those of the glass container industry. Secondly, if glass sand is produced in North Carolina, L-O-F would be the largest potential customer.

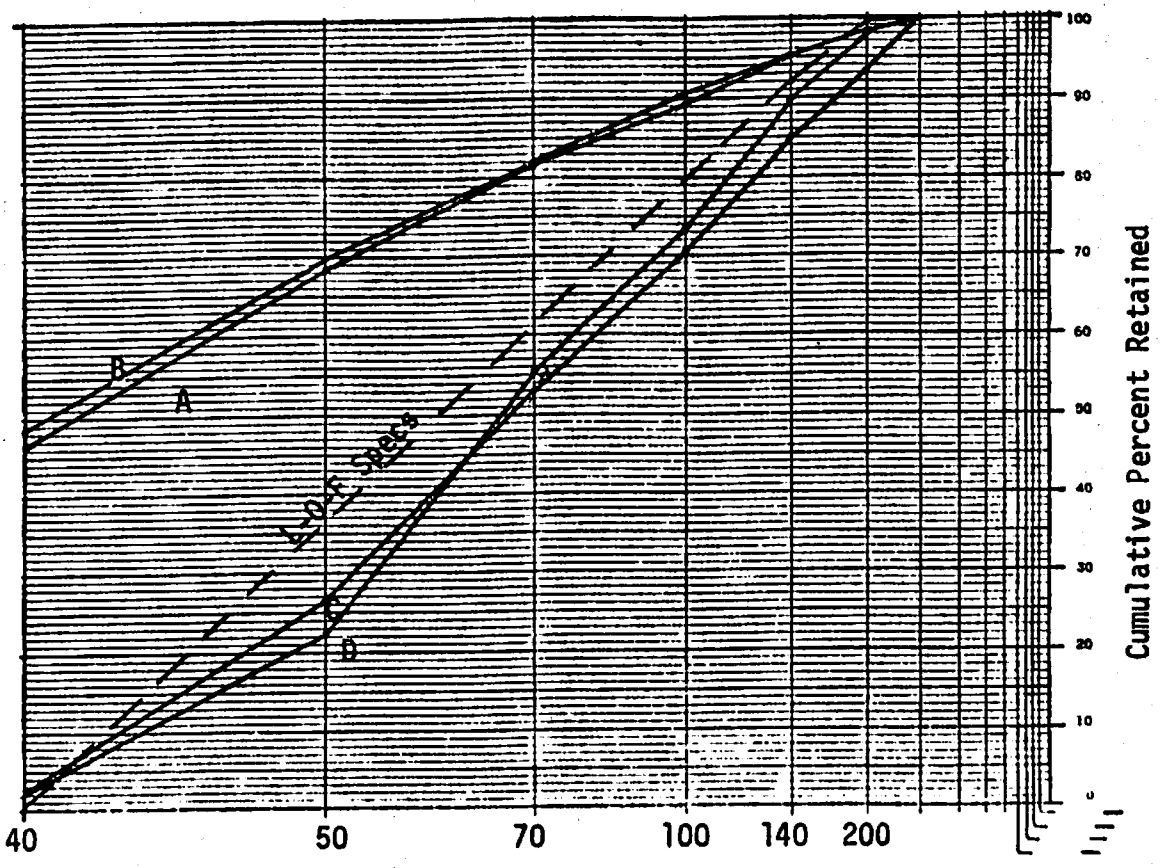
Sizing

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, Figure 1. Approximately 47.0% of the ore was coarser than 40 mesh, and 9% was finer than 140 mesh. The remaining 44% was of intermediate size. Removing the plus-40 mesh fraction produces a product containing an excess of minus-140-mesh material. The minus-140-mesh material could be removed in the desliming stage; however, this would leave a product coarser than



	A		B		C		D	
	Head Feed		+40 M. Removed		+40 & -140 M. Removed		L-O-F Specs	
Mesh U.S.	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights
30	25.0	25.0						
40	21.7	46.7	0.0	0.0	0.0	0.0		0.1
50	19.9	66.6	37.4	37.4	45.5	45.5		
70	12.4	79.0	23.2	60.6	28.2	73.7		
100	6.7	85.7	12.5	73.1	15.3	89.0		
140	4.8	90.5	9.0	82.1	11.0	100.0		92.0
200	2.3	92.8	4.2	86.3				99.5
-200	7.2	100.0	13.7	100.0				100.0
Total	100.0		100.0		100.0			

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



	A		B		C		D	
	Pulper Disch.		Rod Mill Feed		Rod Mill Disch		Silica Prod.	
Mesh U.S.	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights
40	46.8	46.8	48.1	48.1	2.3	2.3	2.0	2.0
50	21.7	68.5	21.8	69.9	24.3	26.6	20.7	22.7
70	13.1	81.6	12.2	82.1	26.4	53.0	32.8	55.5
100	7.7	89.3	8.8	90.9	17.6	70.6	18.0	73.5
140	5.9	95.2	4.8	95.1	14.5	85.1	16.1	89.6
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
Total	100.0		100.0		100.0		100.0	

Figure 2. Screen analyses of products from various stages of pilot plant processing.

that described in L-O-F specifications. A pilot-plant rod-milling test was undertaken to adjust the grind so that deslimed ore could be milled to produce a product approximating the required size specifications, Fig. 2. The charge consisted of eleven 1 1/8" X 32" rods and three 1 3/4" X 32" rods with a total weight of 190 pounds. Screen analyses of products from six pilot-plant tests are shown in the figures in Appendix A.

Pilot Plant Operation

The flowsheet for the pilot plant is shown in Figure 3, and the details of the individual runs are given in the appendices. Approximately 300 pounds of ore per hour were transported by a belt conveyor from a hopper to a pulper. Here water was added and the pulp was agitated for 5 minutes. The material was pumped to a cyclone. The cyclone-overflow slimes discharged to waste, and the underflow discharged to a rod mill. The ore was ground at 24% solids for a retention time of approximately 5 minutes. Rod mill discharge was passed over an integral trommel screen for removal of plus-30-mesh oversize material which was discarded as waste. The screen-undersize material was pumped to a cyclone for desliming. The cyclone-overflow slimes discharged to waste, and the underflow discharged to a spiral classifier for additional slime removal. In the tests where a scrubber was used (tests 8, 9, 10), the cyclone underflow was dewatered preparatory to attrition scrubbing. 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).

Depending on which pilot plant test was being conducted, the scrubber discharge material or the unscrubbed sand product from the first spiral classifier was pumped to a cyclone for desliming. The cyclone-overflow slimes discharged to waste, and the underflow material discharged to a spiral classifier for additional slime removal and dewatering preparatory to conditioning.

The sand product from the classifier was conditioned for approximately 5 minutes at 60 to 65% solids in a pulp containing the reagents being investigated. The material flowed 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 flowed 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.

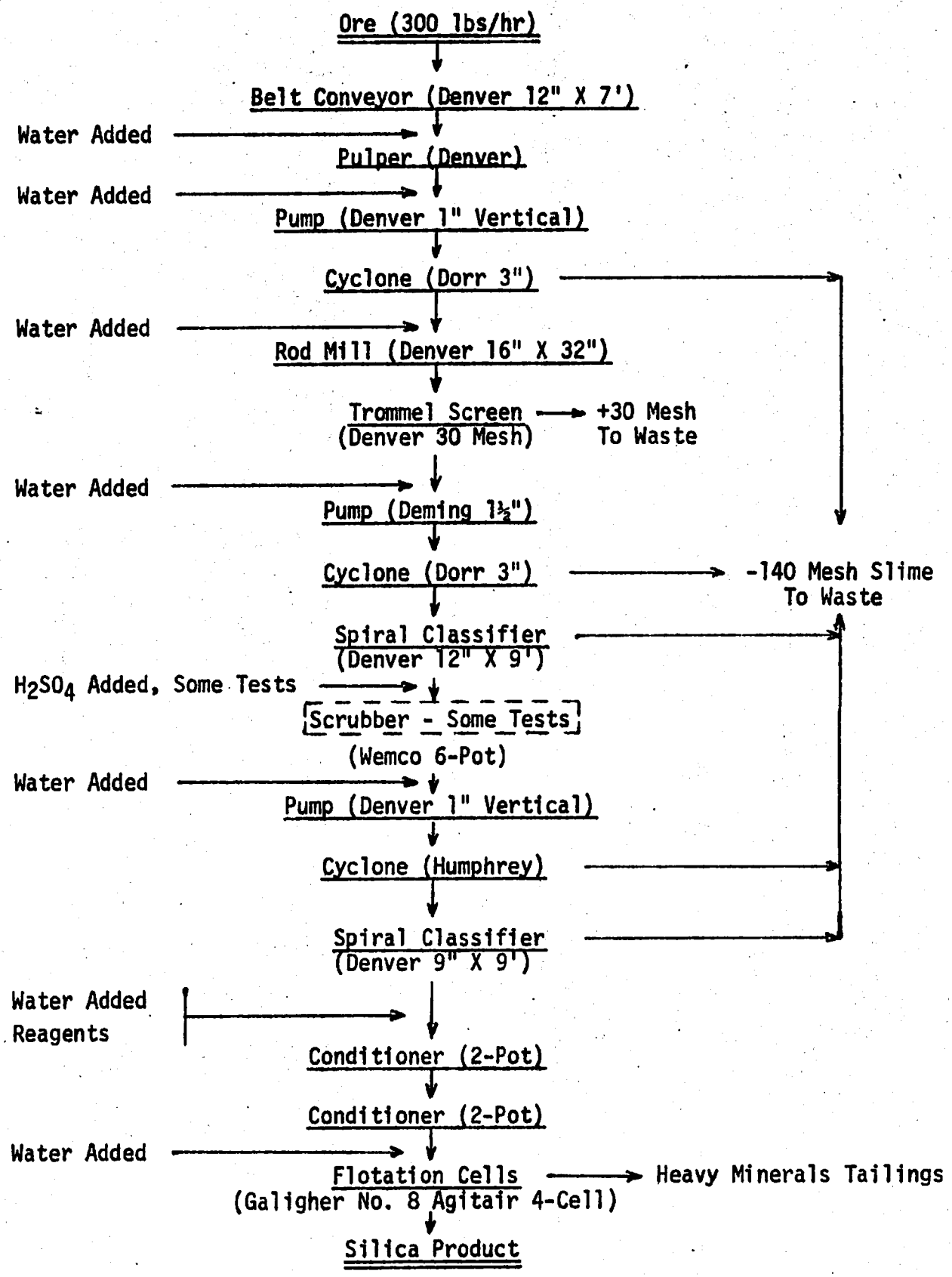


Figure 3. Pilot plant flowsheet

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 of 100-gram samples was used to determine heavy mineral content of feed to flotation and tailings. One hundred gram samples of the silica product was screened on 70 mesh and both fractions were subjected to heavy liquid separation to determine heavy mineral content of sized fractions and total silica product. Each sample contained approximately 50 grams.

It was felt that the amount of heavy minerals obtained from heavy liquid separation of 50 grams of silica product would be so small being in the range of 0.0002 grams that a weighing error might incur. Therefore, in tests 3, 4, 5, 6, 8 and 9, in addition to the above determinations, 2500-gram samples of the bulk silica products were screened on 70 mesh, and the plus and minus fractions were separated in heavy liquid. The heavy-minerals content was determined for both screen fractions and the total product. The heavy minerals were leached for 4 to 5 minutes in a boiling 1:1 solution of HCl and water. Water was added to dilute the HCl, which was then removed by decantation. The heavy minerals were leached with concentrated HNO_3 for 3 minutes, mainly to remove any pyrite which might be present. The refractory heavy minerals were determined by separating the leached heavy minerals with a Frantz Isodynamic Magnetic Separator. The refractory minerals reported to the nonmagnetic product along with some nonrefractory minerals. Grain counts were made of the plus 70 mesh and minus 70 mesh fractions to determine the exact amount of refractory minerals present in the product, and these are the results presented in the tables in the appendices.*

Size distributions of silica products were obtained by screening on a Ro-Tap. Water distribution to equipment was monitored with flowrators, and water consumption was determined with a totalizing meter.

Comparison of L-O-F and MRL Analyses

Fifty pound samples of products from tests 3, 4, 5, 6, 8, and 9 were sent to the Libbey-Owens-Ford Company (L-O-F) laboratory in Toledo, Ohio

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

for evaluation. A comparison of L-O-F data with Minerals Research Laboratory data is shown in Appendix C and explained in the Discussion section.

New Flotation Process

Four of the ten pilot-plant runs used the new heavy-mineral flotation process. The primary feature of this procedure is the use of detergent-type reagents, such as an equal mixture of 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 referred to in the results as Tex-Wet reagent were obtained from Intex Products, Inc., Greenville, South Carolina; however, other brands of similar chemicals were satisfactory in batch tests. The reagent adheres to the contaminant minerals, which are removed in the froth product. Silica is not affected by the collector and is suppressed by the acid media at low pH; hence it is removed at the bottom of the chamber.

Other Flotation Processes

For comparison purposes, a pilot-plant test was performed on the commercial process in which petroleum sulfonate was the heavy-mineral collector. A second, modified commercial process, in which a fatty acid was used as the collector, was also included for comparison purposes. In addition, combinations of these reagents with the experimental detergent-type reagent were evaluated. Pilot plant tests 1 through 7 were run without scrubbing, and test 8, 9, and 10 were run with scrubbing.

RESULTS

The pilot plant tests are described and the results obtained from selected pilot plant tests are given in Appendix A. The first two pilot-plant runs involved equipment adjustment and pilot-plant tune up. Tex-Wet detergent-type reagent was used in both tests, with the lower amount of reagent giving the better grade product; however, the silica products from the first two tests were out of range of the specifications concerning plus-70-mesh refractory heavy minerals; therefore, the test data are not included in the appendix.

The silica produced in test 3, Table I, using fatty acid with the Tex-Wet reagent, contained 0.260 grams of plus-70-mesh refractory heavy minerals per 100 pounds of sand; and the silica yield was 84.3%. The total content of refractory heavy minerals in the silica product was 0.891 grams per 100 pounds of sand.

Petroleum sulfonate was used as the collector in test 4, Table II. This reagent is used commercially for sand beneficiation, and this run was included as a control. In previous research at the Minerals Research Laboratory, the use of petroleum sulfonate as a heavy-minerals collector in sand beneficiation produced the best results. The silica produced in test 4 contained 0.082 grams of plus-70-mesh refractory heavy minerals per 100 pounds of sand, and the silica yield was 74.8%. The total content of refractory-heavy-minerals in the silica product was 1.178 grams per 100 pounds of sand.

The silica produced in test 5, Table III, using Tex-Wet reagent alone, contained 0.527 grams of plus-70-mesh refractory heavy minerals per 100 pounds of sand; and the silica yield was 81.3%. The total content of refractory heavy minerals in the silica product was 0.849 grams per 100 pounds of sand.

The silica produced in test 6, Table IV, in which a reduced amount of petroleum sulfonate was used with the Tex-Wet detergent-type reagent, contained 0.041 grams of plus-70-mesh refractory heavy minerals per 100 pounds of sand; and the silica yield was 83.2%. The total content of refractory heavy minerals in the silica product was 0.545 grams per 100 pounds of sand.

Test 7 involved a process similar to that of a commercial sand company in Camden, Tennessee, in which fatty acid, sodium hydroxide, fuel oil, and pine oil are the reagents. Not only did the sand from this test not meet specifications, but the yield was by far the lowest obtained in the pilot plant experiments -- 62.9%. The test data are not included in the appendix.

The silica produced in test 8, Table V, in which the ore was scrubbed and Tex-Wet reagent used, contained 0.225 grams of plus-70-mesh refractory heavy minerals per 100 pounds of sand; and the silica

yield was 77.0%. The total content of refractory heavy minerals in the silica product was 1.177 grams per 100 pounds of sand.

The silica produced in test 9, Table VI, using scrubbing and fatty acid with Tex-Wet reagent, contained 0.390 grams of plus-70-mesh refractory heavy minerals per 100 pounds of sand; and the silica yield was 84.5%. The total content of refractory heavy minerals in the silica product was 2.680 grams per 100 pounds of sand.

Test 10, using scrubbing and reduced amount of petroleum sulfonate and Tex-Wet reagent, produced sand which did not meet specifications. Data for this test are not given in the appendix.

DISCUSSION

The petroleum sulfonate collector was used as a control because it had produced the best results in the previous sand beneficiation research at the Minerals Research Laboratory. The flotation process using this collector (test 4) produced a silica product with next to the lowest amount of plus-70-mesh refractory heavy minerals, compared with other tests. The product yield for this test was the lowest (74.8%) of all of the tests, and the total content of refractory heavy minerals in the sample was next to the highest. With additional tests, yields in the 80 to 85% range probably could have been obtained with this heavy-minerals collector without exceeding the specifications for iron and refractory minerals in the product.

The flotation process using the anionic detergent-type collector in combination with petroleum sulfonate (test 6) produced a product with a refractory-mineral content similar to that in the petroleum sulfonate test (0.041 vs. 0.082 grams of plus 70 mesh R.H.M.) but with higher (83.2 vs. 74.8%) yield. The total content of refractory heavy minerals in the sample was much less (0.545 vs. 1.178) than that of the test using petroleum sulfonate alone.

The chemical analyses of the products were approximately the same for all tests. The refractory-heavy-minerals contents of the total products varied from 0.545 to 2.680 grams per 100 pounds of sand.

One of the tests (test 5) using the detergent-type reagent produced a product containing the largest amount (0.527 grams per 100 pounds of sand) of plus-70-mesh refractory heavy minerals; however, the amount of total refractory heavy minerals in the product was lower than that of the test using petroleum sulfonate. This would indicate that the process should be adjusted to float more coarse contaminants and thus reduce the plus-70-mesh refractory heavy mienrals in the silica product. Thus there were no significant differences apparent in the results obtained in the pilot plant using petroleum sulfonate collector and those obtained using the anionic detergent-type heavy-minerals collector. This indicates that equivalent results could be obtained with the two reagents in a commercial operation. At current market prices, the anionic detergent-type reagent would cost about $0.05 more per ton of silica produced than would petroleum sulfonate. However, the former is biodegradable while the latter is not. This could be a very important factor in helping to minimize stream pollution problems. With the fluctuating prices and the ever-changing availability of reagents, particularly the petrochemicals, it is good to have several alternatives for removal of contaminant minerals.

The modified commercial process utilizing fatty acid, sodium hydroxide, fuel oil, and pine oil was included because it had not been tested previously at the Laboratory. Under the conditions of these tests, this procedure was not satisfactory because the products did not meet specifications, and the yield was the lowest obtained.

The Tex-Wet detergent-type reagent produces a voluminous froth which appears excessive; however, the froth dissipates rapidly as observed in the pilot plant and in a commercial plant at Camden, Tennessee, where this reagent was field tested. An absence of a good froth was experienced when using petroleum sulfonate or fatty acid (Pamak-25) as collector. However, a good froth was obtained by using the detergent-type reagent in combination with either of the other two reagents.

Under the conditions used, scrubbing was not required to produce silica meeting specifications. In fact, scrubbing seemed to have a detrimental effect.

A comparison of the analyses of MRL with L-O-F showed a slightly higher heavy minerals content for MRL. The MRL refractory heavy minerals content was lower for the plus 70 mesh fraction and higher for the minus 70 mesh fraction than L-O-F. The total refractory heavy minerals in the

product was comparable between the two laboratories. Although the refractory heavy minerals content in the products were marginal or slightly higher than L-O-F specifications, the primary objective of comparing the effect of various reagents for removal of contaminant minerals was accomplished.

Libbey-Owens-Ford Company data is shown in Appendix D.

CONCLUSIONS

The following conclusions were reached:

1) Under the conditions of these tests, silica meeting chemical specifications for use in flat-glass production, and marginal as to limitations of plus-70-mesh refractory minerals can be produced by using a biodegradable anionic detergent-type collector for the heavy minerals.

2) In pilot plant operation, the above collector was nearly as effective alone and just as effective in combination with fatty acid or petroleum sulfonate, as petroleum sulfonate alone. Petroleum sulfonate was the best heavy-minerals collector found in previous sand beneficiation research at the Minerals Research Laboratory.

3) Under the conditions evaluated, scrubbing was not necessary and actually may produce undesirable effects.

4) The modified commercial process utilizing fatty acid, sodium hydroxide, fuel oil, and pine oil is not satisfactory under the conditions evaluated in this research.

RECOMMENDATION

It is likely that sand meeting specifications for use in flat-glass production could be produced by utilizing anionic detergent reagents if more extensive pilot plant tests were conducted. This is recommended if the use of rapidly biodegradable reagents becomes necessary.

APPENDIX A

PILOT PLANT TEST DATA

Test No. 1

This test involved the use of crowder plates in flotation 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[1] per ton of ore were added to the second set of conditioners.

The product contained too much (5.46 grams) plus-70-mesh refractory minerals. Detailed data for test 1 are not given in this report.

Test No. 2

This test was similar to test 1 except the collector was reduced to 1.0 pound per ton of ore and was added to the second set of conditioners instead of the first conditioners. The silica product was better than in the previous test but still contained too much (1.35 grams) plus-70-mesh refractory minerals. Crowder plates may be detrimental, possibly causing bubbles to burst prematurely and release entrapped heavy minerals contaminants. The plates were removed before test 3. Detailed data for test 2 are not given in this report.

Test No. 3

One-half pound of fatty acid [2] and one-half pound of Tex-Wet 1158-1197 per ton of ore were added to the second set of conditioners. Sulfuric acid was added to the first conditioners. Crowder plates had been removed before this test. The silica contained 0.891 grams of refractory minerals per 100 pounds of sand, with 0.260 grams being in the plus-70-mesh fraction. Test 3 data are shown in Table I.

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

[2] Pamak-25 obtained from Hercules, Inc., Wilmington, Delaware.

TABLE I

PILOT PLANT TEST NO. 3

	Physical Data				Chemical Analyses, %				
			Grams per 100 lbs Sand						
Sample	Wt %	% HM	HM	RHM	Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	0.2								
H.M. Float	2.3	23.5			4.30	1.10	-	-	-
-140 Slime	13.2								
+70 M. Silica	51.1	0.0026	0.613	0.260					
-70 M. Silica	48.9	0.0059	1.314	0.631					
Total Silica	84.3	0.0042	1.927	0.891	0.024	0.005	0.008	0.012	0.05
Ore Feed	100.0	0.55							
Flot. Feed	86.5	0.56			0.170	0.460	0.020	0.040	0.05

	Conditions			Reagents (lbs per ton of ore)		
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 Class.	6.0	70				
#3 Pump	-	10				
#3 Cyclone U'flow	-	19				
#2 Spiral Class.	6.0	70				
#1 Conditioner	5.1	58	2.04	1.0	-	-
#2 Conditioner	6.4	58	2.25	-	0.5	0.5
Flot. 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.

Test No. 4

This was a commercial procedure which was included for comparative purposes. Two and one-half pounds of sulfuric acid per ton of ore were added to the first conditioners. One pound of petroleum sulfonate[1] and 0.10 pound of frother[2] per ton of ore were added to the second conditioners. The silica product contained 1.178 grams of refractory minerals per 100 pounds of sand, with 0.082 grams being in the plus-70-mesh fraction. Test 4 data are shown in Table II.

Test No. 5

One pound of sulfuric acid and one pound of Tex-Wet 1158-1197 (see footnote test 1) per ton of ore were added to the first conditioner. The silica product contained 0.849 grams of refractory minerals per 100 pounds of sand, with 0.527 grams being in the plus-70-mesh fraction. Test 5 data are shown in Table III.

Test No. 6

Two and one-half pounds of sulfuric acid per ton of ore were added to the 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 the second conditioners. The silica product contained 0.545 grams of refractory minerals per 100 pounds of sand, with 0.041 grams being in the plus-70-mesh fraction. Detailed data for test 6 are shown in Table IV.

Test No. 7

This test was intended to duplicate a processing technique used in a commercial sand plant in Tennessee, and it was included for comparative purposes. 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. Detailed data for test 7 are not given in this report.

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[1] M-70 obtained from Hunt Chemicals, Marion, North Carolina.

[2] F-75 glycol frother obtained from American Cyanamid Co., Wayne, N.J.

TABLE II

PILOT PLANT TEST NO. 4

	Physical Data				Chemical Analyses, %				
			Grams per 100 lbs Sand						
Sample	Wt %	% HM	HM	RHM	Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	2.0								
H.M. Float	9.3	5.59			0.88	0.84	-	-	-
-140 Slime	13.9								
+70 M. Silica	57.0	0.0016	0.424	0.082					
-70 M. Silica	43.0	0.0148	2.892	1.096					
Total Silica	74.8	0.0073	3.316	1.178	0.017	0.06	0.007	0.006	0.04
Ore Feed	100.0	0.52							
Flot. Feed	84.1	0.52			0.140	0.11	0.02	0.02	0.09

	Conditions			Reagents(lbs per ton of ore)		
Process	Time (min)	% Solids	pH	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	-	10				
#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
Flot. Cells	2.5	11	2.66	-	-	-

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

TABLE III

PILOT PLANT TEST NO. 5

	Physical Data				Chemical Analyses, %				
			Grams per 100 lbs Sand						
Sample	Wt %	% HM	HM	RHM	Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	3.8								
H.M. Float	1.5	22.8			4.10	1.23	-	-	-
-140 Slime	13.4								
+70 M. Silica	51.8	0.0073	1.722	0.527					
-70 M. Silica	48.2	0.0034	0.747	0.322					
Total Silica	81.3	0.0054	2.469	0.849	0.022	0.06	0.006	0.010	0.06
Ore Feed	100.0	0.35							
Flot. Feed	82.8	0.48			0.15	0.460	0.015	0.025	0.09

	Conditions			Reagents(lbs per ton of ore)	
Process	Time (min)	% Solids	pH	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 Class.	5.5	70			
#3 Pump	-	10			
#3 Cyclone U'flow	-	19			
#2 Spiral Class.	5.5	70			
#1 Conditioner	5.4	60	1.8	1.0	1.0
#2 Conditioner	6.7	60	1.9		
Flot. Cells	2.2	12	2.7		

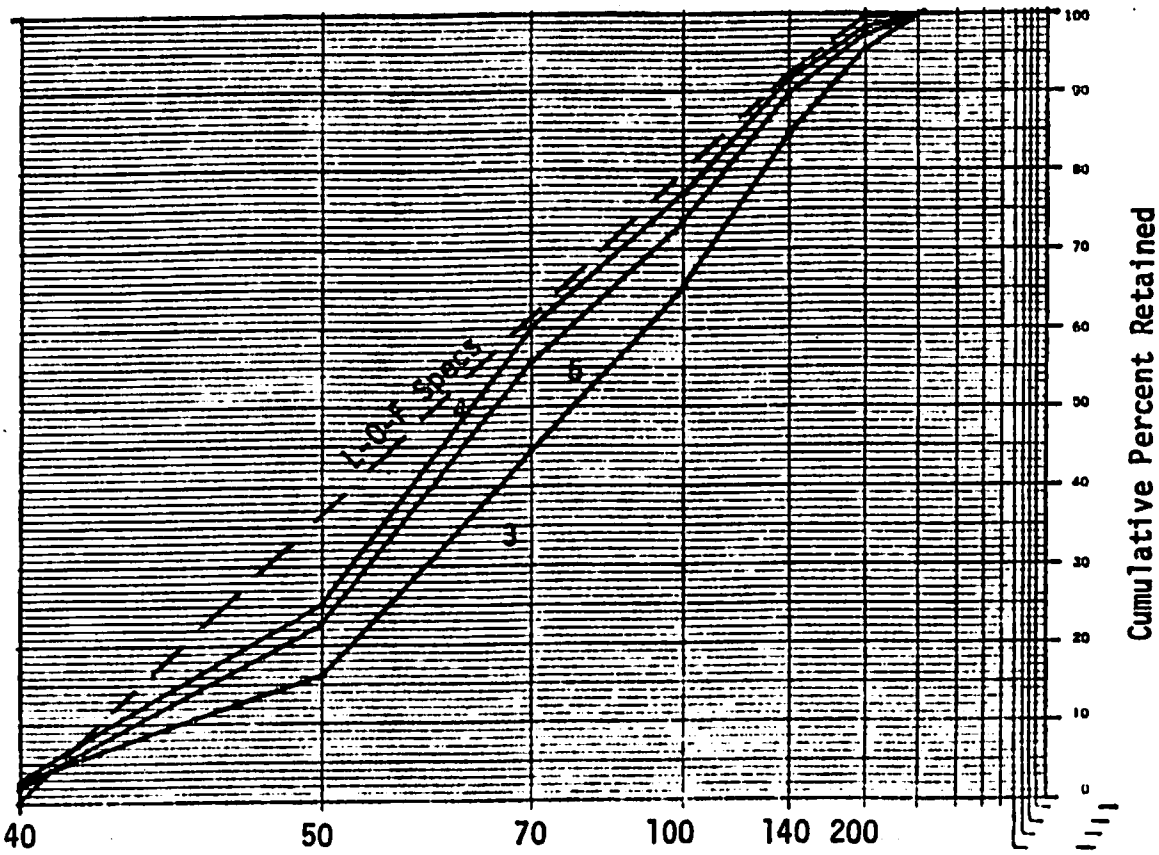
Remarks: Feed Rate - 338 pounds per hour (dry basis).
Water Used - 11,877 gallons per ton of ore.
Collector reagent added to first pot in #1 conditioner.

TABLE IV

PILOT PLANT TEST NO. 6

	Physical Data				Chemical Analyses, %				
			Grams per 100 lbs Sand						
Sample	Wt %	% HM	HM	RHM	Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	1.3								
H.M. Float	2.1	16.2			3.00	1.00	-	-	-
-140 Slime	13.4								
+70 M. Silica	57.9	0.0014	0.369	0.041					
-70 M. Silica	42.1	0.0064	1.226	0.504					
Total Silica	83.2	0.0035	1.595	0.545	0.023	0.07	0.005	0.007	0.11
Ore Feed	100.0	0.35							
Flot. Feed	85.3	0.43			0.16	0.50	0.02	0.04	0.24

	Conditions			Reagents(lbs per ton of ore)		
Process	Time (min)	% Solids	pH	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 Class.	5.4	70				
#3 Pump	-	8				
#3 Cyclone U'flow	-	13				
#2 Spiral Class.	5.4	70				
#1 Conditioner	5.5	63	1.75	2.5		
#2 Conditioner	6.9	63	2.04		0.5	0.5
Flot. Cells	2.3	13	2.47			



	L-O-F Specs		Test 3		Test 4		Test 5	
Mesh	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights
40		0.1	2.5	2.5	2.5	2.5	2.5	2.0
50			13.7	16.2	22.7	25.2	20.7	22.7
70			28.2	44.4	34.9	60.1	32.8	55.5
100			20.8	65.2	17.0	77.1	18.0	73.5
140		92.0	19.6	84.8	14.8	91.9	16.1	89.6
200		99.5	10.6	95.4	6.6	98.5	8.1	97.7
-200		100.0	4.6	100.0	1.5	100.0	2.3	100.0
Total			100.0		100.0		100.0	

Figure 4. Screen analyses - pilot plant products.

Test No. 8

An attrition scrubber was used in this test for additional cleaning of mineral grains for reagent attachment. Two pounds of sulfuric acid per ton of ore were 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 1.177 grams of refractory minerals per 100 pounds of sand, with 0.225 grams being in the plus-70-mesh fraction. Detailed data for test 8 are shown in Table V.

Test No. 9

An attrition scrubber was used in this test for additional cleaning of mineral grains for reagent attachment. Two pounds of sulfuric acid per ton of ore were fed to the scrubber. One pound of sulfuric acid, 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 2.680 grams of refractory minerals per 100 pounds of sand, with 0.390 grams being in the plus-70-mesh fraction. Detailed data for test 9 are shown in Table VI.

Test No. 10

An attrition scrubber was used in this test for additional cleaning of mineral grains for reagent attachment. A considerably reduced reagent charge was employed for this run. Two pounds of sulfuric acid per ton of ore were fed to the scrubber. One pound of sulfuric acid, 0.2 pound of petroleum sulfonate (M-70), and 0.2 pound of Tex-Wet 1158-1197 were added to the first conditioner. The silica product contained an excessive amount of plus-70-mesh refractory minerals. Detailed data for test 10 are not given in this report.

TABLE V

PILOT PLANT TEST NO. 8

Sample	Physical Data Wt %	% HM	Grams per 100 lbs Sand HM	RHM	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 Slime	13.1								
+70 M. Silica	55.4	0.0069	1.732	0.225					
-70 M. Silica	44.6	0.0155	3.131	0.952					
Total Silica	77.0	0.0107	4.863	1.177	0.027	0.044	0.0062	0.0061	0.10
Ore Feed	100.0	0.357							
Flot. Feed	83.3	0.369			0.114	0.41	0.0184	0.0312	0.12

Process	Conditions Time (min)	% Solids	Reagents(lbs per ton of ore) H_2SO_4	TW-1197 TW-1158
Belt Feeder	-	-		
Pulper	-	-		
#1 Pump	-	-		
#1 Cyclone U'flow	-	-		
Rod Mill	-	-		
Trommel Screen	-	-		
#2 Pump	-	-		
#2 Cyclone U'flow	-	-		
#1 Spiral Classifier	-	-		
Scrubber	-	-	2.0	-
#3 Pump	-	-		
#3 Cyclone U'flow	-	-		
#2 Spiral Classifier	-	-		
#1 Conditioner	-	-	1.0	1.0
#2 Conditioner	-	-		
Flot. Cells	-	-		

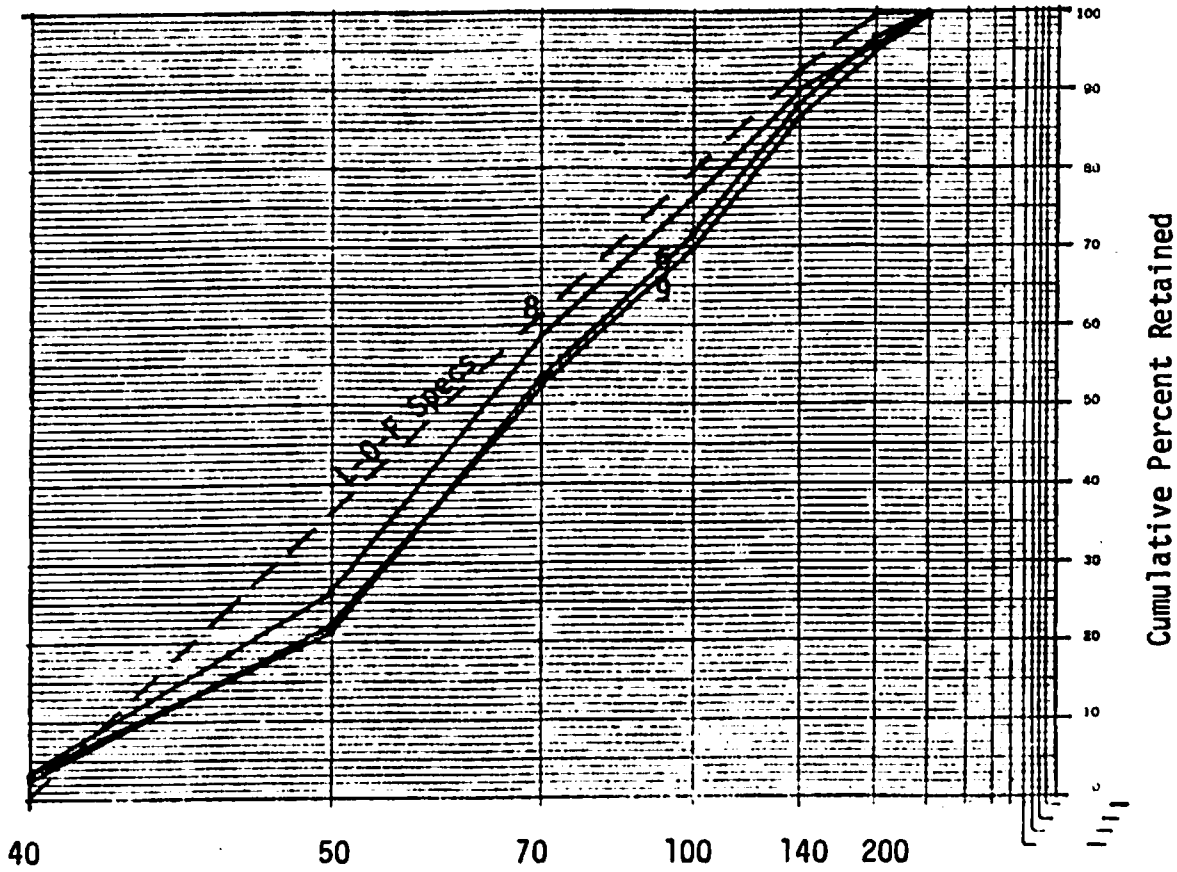
TABLE VI

PILOT PLANT TEST NO. 9

	Physical Data				Chemical Analyses, %				
			Grams per 100 lbs Sand						
Sample	Wt %	% HM	HM	RHM	Fe_2O_3	Al_2O_3	Na_2O	K_2O	LOI
+30 Mesh	2.4								
H.M. Float	2.0	18.8			3.50	0.80	-	-	-
-140 Slime	11.1								
+70 M. Silica	48.6	0.0078	1.714	0.390					
-70 M. Silica	51.4	0.0277	6.487	2.290					
Total Silica	84.5	0.0181	8.201	2.680	0.0270	0.039	0.0054	0.0034	0.04
Ore Feed	100.0	0.40							
Flot. Feed	86.5	0.44			0.17	0.398	0.0140	0.0184	0.15

	Conditions			Reagents(lbs per ton of ore)		
Process	Time (min)	% Solids	pH	H_2SO_4	Pamak 25	TW-1197 TW-1158
Belt Feeder	-					
Pulper	-	13				
#1 Pump	-	-				
#1 Cyclone U'flow	-	22				
Rod Mill	-	28				
Trommel Screen	-	-				
#2 Pump	-	-				
#2 Cyclone U'flow	-	15				
#1 Spiral Classifier	-	-				
Scrubber	-	70	3.2	2.0		
#3 Pump	-	-				
#3 Cyclone U'flow	-	-				
#2 Spiral Classifier	-	-				
#1 Conditioner	-	25	2.4	1.0	0.4	0.2
#2 Conditioner	-	4				
Flot. Cells	-	-				

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



	L-O-F Specs		Test 6		Test 8		Test 9	
Mesh	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights	Per Cent	Per Cent Cumulative Weights
40		0.1	3.0	3.0	3.2	3.2	2.9	2.9
50			18.4	21.4	23.2	26.4	19.1	22.0
70			31.8	53.2	32.4	58.8	30.3	52.3
100			18.2	71.4	17.3	76.1	17.5	69.8
140		92.0	16.6	88.0	13.5	89.6	16.4	86.2
200		99.5	8.6	96.6	6.3	95.9	8.8	95.0
-200		100.0	3.4	100.0	4.1	100.0	5.0	100.0
Total			100.0		100.0		100.0	

Figure 5. Screen analyses - pilot plant silica products.

APPENDIX B

PRODUCT SUMMARY DATA

Test No.	Reagent	% Yield	Total Heavy Minerals Grams per 100 lbs Sand HM	Total Heavy Minerals Grams per 100 lbs Sand RHM	+70 Mesh Heavy Minerals Grams per 100 lbs Sand HM	+70 Mesh Heavy Minerals Grams per 100 lbs Sand RHM
3	Tex-Wet Pamak 25	84.3	1.927	0.891	0.613	0.260
4	M-70 F-75	74.8	3.316	1.178	0.424	0.082
5	Tex-Wet	81.3	2.469	0.849	1.722	0.527
6	M-70 Tex-Wet	83.2	1.595	0.545	0.369	0.041
8	Tex-Wet	77.0	4.863	1.177	1.732	0.225
9	Pamak 25 Tex-Wet	84.5	8.201	2.680	1.714	0.390

Test No.	% Fe_2O_3	% Al_2O_3	% Na_2O	% K_2O	% LOI
3	0.024	0.005	0.008	0.012	0.05
4	0.017	0.060	0.007	0.006	0.04
5	0.022	0.060	0.006	0.010	0.06
6	0.023	0.070	0.005	0.007	0.11
8	0.027	0.044	0.006	0.006	0.10
9	0.027	0.039	0.005	0.003	0.04

APPENDIX C

COMPARISON OF L-O-F AND MRL ANALYSES

(100 lb Sample Basis)

L-O-F Tests used 25 lbs

MRL Tests used 2500 grams or 5.51 lbs

Test No.	Heavy Minerals Wt (Grams)				Percent Heavy Minerals			
	Untreated		Acid Treated		Untreated		Acid Treated	
	L-O-F	MRL	L-O-F	MRL	L-O-F	MRL	L-O-F	MRL
3	2.736	2.494	1.628	1.927	0.0060	0.0055	0.0036	0.0042
4	3.536	3.840	2.652	3.316	0.0078	0.0085	0.0058	0.0073
5	3.844	3.893	2.260	2.470	0.0085	0.0086	0.0050	0.0054
6	2.620	2.428	1.324	1.594	0.0058	0.0053	0.0029	0.0035
8	5.644	6.630	3.704	4.862	0.0124	0.0146	0.0082	0.0107
9	9.068	9.253	7.068	8.201	0.0200	0.0204	0.0156	0.0181

REFRACTORY CONTENT

(Grams per 100 lbs Sand)

L-O-F Microscopic Analyses (Grain Count)

MRL Frantz Isodynamic Magnetic Separator and Microscopic Analyses (Grain Count)

Test No.	+40 Mesh		-40+70 Mesh		Cum. +70		-70 Mesh		Total	
	L-O-F	MRL	L-O-F	MRL	L-O-F	MRL	L-O-F	MRL	L-O-F	MRL
3	0.1372		0.4526		0.5898	0.260	0.5434	0.631	1.1332	0.891
4	-		-		-	0.082	-	1.096	-	1.178
5	-		-		-	0.527	-	0.322	-	0.849
6	0.0364		0.2464		0.2828	0.041	0.1059	0.504	0.3887	0.545
8	0.0300		0.5240		0.5540	0.225	0.4356	0.952	0.9896	1.177
9	0.0400		0.7120		0.7520	0.390	2.5670	2.290	3.3190	2.680
Model Sand Spec.:					0.200					

APPENDIX D

L-O-F DATA

LIBBEY-OWENS-FORD COMPANY

TECHNICAL CENTER
1701 EAST BROADWAY, TOLEDO, OHIO 43605
telephone (419) 247-3731

February 24, 1975

Mr. Robert M. Lewis
Senior Mineral Dressing Engineer
Minerals Research Laboratory
North Carolina State University
180 Coxe Avenue
Asheville, North Carolina 28861

Dear Mr. Lewis:

The group of six pilot plant samples of processed sand have been subjected to a broad spectrum of analyses. The complete report issued by the Libbey-Owens-Ford Analytical Control Laboratory is appended. The chemistry of these samples is rated excellent, and should be highly acceptable to the three major divisions of the Glass Industry -- fiber glass, container glass, and flat glass. It should be noted that data on trace colorants have been omitted. Our investigative studies of the surficial sand deposits in the Sand Hills District of eastern North Carolina indicate that cobalt, chromium, nickel, and manganese are present at insignificant levels.

Overall, the mesh distribution would be very acceptable for both flat glass and container glass operations. On the coarse end, LOF would require removal of the plus 40-mesh fraction. This is a precautionary measure applied only to those sands requiring froth flotation. On the fine end, there is concern about dust. It is preferred that the cumulative retained on 200-mesh be not less than 99.7%, with 100.0% retained on 325-mesh.

The heavy mineral loading in the Sand Hills District sands is known to range up to 500 grams per hundred pounds of raw sand. The pilot flotation operations have effectively reduced the residual heavy mineral loadings in each of the six samples.

Very truly yours,

LIBBEY-OWENS-FORD CO.

C. Justus Brown, Jr.
Senior Chemical Engineer
Glass Batch Materials
and Operation

CJB/lh
Enclosure
cc- Mr. J. W. Blumer, LOF
Dr. H. R. Swift, "
Mr. R. R. Snow, "
Mr. E. H. Cunningham, "

LIBBEY-OWENS-FORD COMPANY
TOLEDO CONTROL LABORATORY REPORT

Date: 2/17/75
Copies: J. W. Blumer
H. R. Swift
R. R. Snow
C. J. Brown
R. A. Stevens
Research File

Purchasing (3)

FINAL REPORT

Kind of Material	Sand (Pilot Plant Processed) Purchasing
Shipped by	Carolina Silica Company By:N.C.S.U. Mineralogical Labs.
Shipped from	Asheville, N. C.
Car No.	
Sample consisted of	6 - Samples respectively marked: RML-3, RML-4, RML-5,
Received by Laboratory	12-9-74 RML-6, RML-8, & RML-9.
Laboratory Number	TP-74-358 thru 363 Submitted by C. J. Brown
Work Request No.	CA-74-234

Analysis

Lab. No.	TP-74-358	TP-74-359	TP-74-360	TP-74-361	TP-74-362	TP-74-363
Sample No.	RML-3	RML-4	RML-5	RML-6	RML-8	RML-9
Chem. Anal. (As Rec'd.)						
Ignition Loss	.04	.06	.07	.06	.02	.05
(Moisture)	(.03)	(.03)	(.03)	(.02)	(.02)	(.02)
SiO_2 (by diff.)	99.84	99.82	99.83	99.61	99.86	99.84
Fe_2O_3	.019	.015	.018	.020	.020	.022
Al_2O_3	.086	.088	.071	.069	.087	.068
TiO_2	.010	.010	.009	.011	.013	.015
CaO	.00	.00	.00	.00	.00	.00
MgO	.00	.00	.00	.00	.00	.00
Na_2O	.00	.00	.00	.20	.00	.00
K_2O	.00	.00	.00	.01	.00	.00
SO_3	.00	.01	.00	.02	.00	.00
Total	100.00	100.00	100.00	100.00	100.00	100.00
U.S.Mesh(% Cum.)						
12	None	None	None	None	None	None
16	None	None	None	None	None	3 pcs.
20	None	None	None	None	None	Trace
30	.1	.1	Trace	.1	.1	.1
40	3.2	3.0	2.3	3.5	3.3	3.3
50	24.6	28.0	22.2	30.8	27.3	25.1
70	52.1	59.2	51.8	59.2	56.1	53.5
100	74.5	81.2	75.5	80.1	77.7	75.6
140	88.2	92.7	89.8	91.9	89.8	88.3
200	96.3	98.4	97.7	98.0	96.4	95.7
270	98.8	99.9	99.6	99.7	98.7	98.7
325	99.6	100.0	99.9	99.9	99.6	99.7
(-325)	(.4)	(Trace)	(.1)	(.1)	(.4)	(.3)

Results of Microscopic Examination of HCl & HNO_3 Treated Heavy Minerals

Separated from Carolina Silica Company Sand Processed by N.C.S.U. Mineralogical Labs.

Sample No. RML-3, Lab. No. TP-74-358

Heavy Minerals - 25.0 lb. Test Sample

Mineral Description	In Sample Weight	Per 100 lbs. Weight	Per 100 lbs. Percent
Total heavy minerals.	.684 gms.	2.736 gms.	.0060
HCl & HNO_3 treated heavy minerals.	.407 gms.	1.628 gms.	.0036

Estimated Percentages of HCl & HNO_3 Treated Mineral Constituents, by Mesh Sizes

	+40 Mesh (.049 gm.)		-40+70 Mesh (.164 gm.)		-70+100 Mesh (.077 gm.)		-100 Mesh (.177 gm.)		Overall Estimates	
Mineral Constituent	% of Fraction	% of Treated Minerals	% of Fraction	% of Treated Minerals	% of Fraction	% of Treated Minerals	% of Fraction	% of Treated Minerals	% of Treated Minerals	% of Test Sample
Chromite	7	.84	4	1.61	Trace	Trace	2	.58	3.03	.0001
Zircon	-	-	Trace	Trace	Trace	Trace	3	.86	.86	.0000
Kyanite	60	7.23	45	18.13	35	6.62	30	8.62	40.60	.0015
Andalusite	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Corundum	-	-	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Spinel	-	-	Rare	Rare	-	-	-	-	Rare	Rare
Staurolite	3	.36	20	8.06	15	2.84	20	5.75	17.01	.0006
Ferrosilicates	30	3.61	30	12.09	20	3.78	15	4.31	23.79	.0009
Leucoxene	-	-	1	.40	30	5.68	30	8.63	14.71	.0005
Rutile	-	-	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Clinoenstatite	Trace	Trace	Trace	Trace	Trace	Trace	-	-	Trace	Trace
		12.04		40.29		18.92		28.75	100.00	.0036

Refractory Content

	Grams Refractory Per 100 lbs. of Sample, by Mesh Sizes						Percentage of
Mineral Constituent	+40	-40+70	+70 Cum.	-70+100	-100	Total	Total Refractory
Chromite	.0137	.0262	.0399	Trace	.0142	.0541	4.78
Zircon	-	Trace	Trace	Trace	.0212	.0212	1.87
Kyanite	.1176	.2952	.4128	.1078	.2124	.7330	64.68
Andalusite	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Corundum	-	Trace	Trace	Trace	Trace	Trace	Trace
Spinel	-	Rare	Rare	-	-	Rare	Rare
Staurolite	.0059	.1312	.1371	.0462	.1416	.3249	28.67
	.1372	.4526	.5898	.1540	.3894	1.1332	100.00
Model Sand Spec.	-	-	.200 gm. Max.	-	-	-	-

Sample No. RML-4, Lab. No. TP-74-359

Heavy Minerals - 25.0 lb. Test Sample

Mineral Description	In Sample Weight	Per 100 lbs. Weight	Per 100 lbs. Percent
Total heavy minerals.	.884 gm.	3.536 gms.	.0078
HCl & HNO_3 treated heavy minerals.	.663 gm.	2.652 gms.	.0058

Sample No. RML-5, Lab. No. TP-74-360

Heavy Minerals - 25.0 lb. Test Sample

Mineral Descriptions	In Sample Weight	Per 100 lbs. Weight	Per 100 lbs. Percent
Total heavy minerals.	.961 gm.	3.844 gms.	.0085
HCl & HNO_3 treated heavy minerals	.565 gm.	2.260 gms.	.0050

Results of Microscopic Examination of HCl & HNO_3 Treated Heavy Minerals Separated from Carolina Silica Company Sand Processed by N.C.S.U. Mineralogical Labs

Sample No. RML-6, Lab. No. TP-74-360

Heavy Minerals - 25.0 lb. Test Sample

Mineral Constituent	In Sample Weight	Per 100 lbs. Weight	Per 100 lbs. Percent
Total heavy minerals.	.655 gm.	2.6200 gms.	.0058
HCl & HNO_3 treated heavy minerals.	.331 gm.	1.3240 gms.	.0029

Estimated Percentages of HCl & HNO_3 Treated Mineral Constituents, by Mesh Sizes

Mineral Constituent	+40 Mesh (.013 gm.) % of Fraction	+40 Mesh (.013 gm.) % of Treated Minerals	-40+70 Mesh(.154 gm.) % of Fraction	-40+70 Mesh(.154 gm.) % of Treated Minerals	-70+100 Mesh(.094 gm.) % of Fraction	-70+100 Mesh(.094 gm.) % of Treated Minerals	-100 Mesh(.070 gm.) % of Fraction	-100 Mesh(.070 gm.) % of Treated Minerals	Overall Estimates % of Treated Minerals	Overall Estimates % of Test Sample
Chromite	---	---	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Zircon	---	---	Trace	Trace	Trace	Trace	4	.85	.85	.0000
Kyanite	60	2.36	35	16.28	15	4.26	10	2.11	25.01	.0007
Andalusite	---	---	Trace	Trace	Trace	Trace	---	---	Trace	Trace
Corundum	---	---	Trace	Trace	---	---	---	---	Trace	Trace
Staurolite	10	.39	5	2.33	2	.57	1	.21	3.50	.0001
Ferrosilicates	15	.59	20	9.30	3	.85	5	1.06	11.80	.0004
Leucoxene	15	.59	40	18.61	80	22.72	80	16.92	58.84	.0017
Rutile	---	---	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Clinoenstatite	1 pc.	Rare	Trace	Trace	Trace	Trace	---	---	Trace	Trace
Forsterite	1 pc.	Rare	---	---	---	---	---	---	Rare	Rare
Al-Refractory	1 pc.	Rare	---	---	---	---	---	---	Rare	Rare
		3.93		46.52		28.40		21.15	100.00	.0029

Refractory Content

Grams Refractory Per 100 lbs. of Sample, by Mesh Sizes

Mineral Constituent	+40	-40+70	+70 Cum.	-70+100	-100	Total	Percentage of Total Refractory
Chromite	---	Trace	Trace	Trace	Trace	Trace	Trace
Zircon	---	Trace	Trace	Trace	.0112	.0112	2.88
Kyanite	.0312	.2156	.2468	.0564	.0280	.3312	85.21
Andalusite	---	Trace	Trace	Trace	---	Trace	Trace
Corundum	---	Trace	Trace	---	---	Trace	Trace
Staurolite	.0052	.0308	.0360	.0075	.0028	.0463	11.91
Al-Refractory	4 pcs.	---	4 pcs.	---	---	4 pcs.	Rare
	.0364	.2464	.2828	.0639	.0420	.3887	100.00
Model Sand Spec.	---	---	.200 gm. Max.	---	---	---	---

Results of Microscopic Examination of HCl & HNO_3 Treated Heavy Minerals Separated from Carolina Silica Company Sand Processed by N.C.S.U. Mineralogical Labs

Sample No. RML-8, Lab. No. TP-74-362
Heavy Minerals - 25.0 lb. Test Sample

Mineral Constituent	In Sample Weight	Per 100 lbs. Weight	Per 100 lbs. Percent
Total heavy minerals.	1.411 gms.	5.644 gms.	.0124
HCl & HNO_3 treated heavy minerals.	.926 gm.	3.704 gms.	.0082

Estimated Percentages of HCl & HNO_3 Treated Mineral Constituents, by Mesh Sizes

Mineral Constituent	+40 Mesh (.015 gm.) % of Fraction	+40 Mesh (.015 gm.) % of Treated Minerals	-40+70 Mesh(.397 gm.) % of Fraction	-40+70 Mesh(.397 gm.) % of Treated Minerals	-70+100 Mesh (.293 gm.) % of Fraction	-70+100 Mesh (.293 gm.) % of Treated Minerals	-100 Mesh(.221 gm.) % of Fraction	-100 Mesh(.221 gm.) % of Treated Minerals	Overall Estimates % of Treated Minerals	Overall Estimates % of Test Sample
Chromite	---	---	Trace	Trace	---	---	---	---	Trace	Trace
Zircon	---	---	Trace	Trace	1	.31	20	4.77	5.08	.0004
Kyanite	50	.81	30	12.86	5	1.58	5	1.19	16.44	.0013
Andalusite	---	---	Trace	Trace	Trace	Trace	---	---	Trace	Trace
Staurolite	1 pc.	Rare	3	1.29	1	.32	15	3.58	5.19	.0004
Ferrosilicates	40	.65	15	6.43	8	2.53	10	2.39	12.00	.0010
Leucoxene	10	.16	50	21.43	85	26.90	50	11.94	60.43	.0050
Rutile	1 pc.	Rare	2	.86	Trace	Trace	Trace	Trace	.86	.0001
Clinoenstatite	---	---	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
		1.62		42.87		31.64		23.87	100.00	.0082

Refractory Content

Mineral Constituent	Grams Refractory Per 100 lbs. of Sample, by Mesh Sizes +40	-40+70	+70 Cum.	-70+100	-100	Total	Percentage of Total Refractory
Chromite	---	Trace	Trace	---	---	Trace	Trace
Zircon	---	Trace	Trace	.0117	.1768	.1885	19.05
Kyanite	.0300	.4764	.5064	.0586	.0442	.6092	61.56
Andalusite	---	Trace	Trace	Trace	---	Trace	Trace
Staurolite	4 pcs.	.0476	.0476	.0117	.1326	.1919	19.39
	.0300	.5240	.5540	.0820	.3536	.9896	100.00
Model Sand Spec.	---	---	.200 gm. Max.	---	---	---	---

Results of Microscopic Examination of HCl & HNO_3 Treated Heavy Minerals Separated from Carolina Silica Company Sand Processed by N.C.S.U. Mineralogical Labs

Sample No. RML-9, Lab. No. TP-74-363
Heavy Minerals - 25.0 lb. Test Sample

Mineral Constituent	In Sample Weight	Per 100 lbs. Weight	Per 100 lbs. Percent
Total heavy minerals	2.267 gms.	9.068 gms.	.0200
HCl & HNO_3 treated heavy minerals.	1.767 gms.	7.068 gms.	.0156

Estimated Percentages of HCl & HNO_3 Treated Mineral Constituents, by Mesh Sizes

Mineral Constituent	+40 Mesh(.020 gm.) % of Fraction	+40 Mesh(.020 gm.) % of Treated Minerals	-40+70 Mesh(.445 gm.) % of Fraction	-40+70 Mesh(.445 gm.) % of Treated Minerals	-70+100 Mesh(.567 gm.) % of Fraction	-70+100 Mesh(.567 gm.) % of Treated Minerals	-100 Mesh(.735 gm.) % of Fraction	-100 Mesh(.735 gm.) % of Treated Minerals	Overall Estimates % of Treated Minerals	Overall Estimates % of Test Sample
Chromite	---	---	---	---	---	---	---	---	None	---
Zircon	1 pc.	Rare	Trace	Trace	3	.96	35	14.56	15.52	.0024
Kyanite	40	.45	25	6.30	25	8.02	15	6.24	21.01	.0033
Andalusite	1 pc.	Rare	Trace	Trace	Trace	Trace	---	---	Trace	Trace
Corundum	1 pc.	Rare	---	---	---	---	---	---	Rare	Rare
Spinel	---	---	Trace	Trace	Trace	Trace	Trace	Trace	Trace	Trace
Staurolite	10	.11	15	3.78	10	3.22	8	3.33	10.44	.0016
Ferrosilicates	45	.51	30	7.55	15	4.81	10	4.16	17.03	.0027
Leucoxene	5	.06	25	6.29	45	14.44	30	12.48	33.27	.0052
Rutile	1 pc.	Rare	5	1.26	2	.64	2	.83	2.73	.0004
Clinoenstatite	1 pc.	Rare	Trace	Trace	Trace	Trace	---	---	Trace	Trace
Al-Refractory	1 pc.	Rare	---	---	---	---	---	---	Rare	Rare
		1.13		25.18		32.09		41.60	100.00	.0156

Refractory Content

Mineral Constituent	Grams Refractory Per 100 lbs. of Sample, by Mesh Sizes +40	-40+70	+70 Cum.	-70+100	-100	Total	Percentage of Total Refractory
Chromite	---	---	---	---	---	---	None
Zircon	4 pcs.	Trace	Trace	.0680	1.0290	1.0970	33.05
Kyanite	.0320	.4450	.4770	.5670	.4410	1.4850	44.74
Andalusite	4 pcs.	Trace	Trace	Trace	---	Trace	Trace
Corundum	4 pcs.	---	4 pcs.	---	---	4 pcs.	Rare
Spinel	---	Trace	Trace	Trace	Trace	Trace	Trace
Staurolite	.0080	.2670	.2750	.2268	.2352	.7370	22.21
Al-Refractory	4 pcs.	---	4 pcs.	---	---	4 pcs.	Rare
	.0400	.7120	.7520	.8618	1.7052	3.3190	100.00
Model Sand Spec.	---	---	.200 Max.	---	---	---	

Results of Tests for Refractory-Type Particles in Heavy Minerals Separated From Carolina Silica Co. Sand (Pilot Processed by N.C.S.U. Mineralogical Labs.), Treated with HCl & HNO_3, and Dispersed in Regular Glass Melt

Sample No. RML-3, Lab. No. TP-74-358

Mineral Constituent	Least Dimension of Particle	Quantities of Particles Observed Dispersed in Glass Melt: Particles in Melt Heated for 1-Hour @ 2400° F — From 25.0 lb. Sample	Particles in Melt Heated for 1-Hour @ 2400° F — Per 100 lbs.	Particles in Melt Heated for an Additional 1-Hour @ 2600° F — From 25.0 lb. Sample	Particles in Melt Heated for an Additional 1-Hour @ 2600° F — Per 100 lbs.
Chromite	.01"-.02"	7	28	0	0
	.0065"-.01"	59	236	26	104
	.003"-.0065"	17	68	14	56
	< .003"	24	96	8	32
		107	428	48	192
Zircon	.0065"-.01"	6	24	2	8
	.003"-.0065"	24	96	2	8
	<.003"	~ 48	~ 192	0	0
		~ 78	~ 312	4	16
Kyanite	.02"-.03"	20	80	0	0
	.01"-.02"	52	208	10	40
	.0065"-.01"	> 500	> 2,000	9	36
	.003"-.0065"	> 300	>1,200	25	100
	<.003"	> 200	> 800	0	0
		>1,072	> 4,288	44	176
Andalusite	.02"-.03"	1	4	0	0
	.01"-.02"	2	8	0	0
	.0065"-.01"	5	20	3	12
	.003"-.0065"	4	16	0	0
	<.003"	3	12	0	0
		15	60	3	12
Corundum	.01"-.02"	1	4	1	4
	.0065"-.01"	0	0	0	0
	.003"-.0065"	4	16	0	0
	<.003"	0	0	0	0
		5	20	1	4
Spinel	.0065"-.01"	1	4	0	0
	.003"-.0065"	1	4	0	0
	<.003"	0	0	0	0
		2	8	0	0
Staurolite	.01"-.02"	3	12	0	0
	.0065"-.01"	29	116	0	0
	.003"-.0065"	15	60	0	0
	<.003"	0	0	0	0
		47	188	0	0

Results of Tests for Refractory-Type Particles in Heavy Minerals Separated From Carolina Silica Co. Sand (Pilot Processed by N.C.S.U. Mineralogical Labs.), Treated with HCl & HNO_3, and Dispersed in Regular Glass Melt

Sample No. RML-4, Lab. No. TP-74-359

Mineral Constituent	Least Dimension of Particle	Quantities of Particles Observed Dispersed in Glass Melt: Particles in Melt Heated for 1-Hour @ 2400° F — From 25.0 lb. Sample	Particles in Melt Heated for 1-Hour @ 2400° F — Per 100 lbs.	Particles in Melt Heated for an Additional 1-Hour @ 2600°F — From 25.0 lb. Sample	Particles in Melt Heated for an Additional 1-Hour @ 2600°F — Per 100 lbs.
Chromite	.01"-.02"	2	8	1	4
	.0065"-.01"	0	0	2	8
	.003"-.0065"	4	16	1	4
	<.003"	1	4	3	12
		7	28	7	28
Zircon	.0065"-.01"	36	144	3	12
	.003"-.0065"	∽ 200	∽ 800	43	172
	< .003"	∽ 300	∽1,200	>75	> 300
		∽ 536	∽2,144	>121	> 484
Kyanite	.02"-.03"	4	16	3	12
	.01"-.02"	∽ 100	∽ 400	23	92
	.0065"-.01"	∽300	∽1,200	49	196
	.003"-.0065"	∽ 200	∽ 800	> 100	> 400
	<.003"	∽100	∽ 400	5	20
		∽ 704	∽2,816	> 180	> 720
Andalusite	.01"-.02"	3	12	1	4
	.0065"-.01"	12	48	7	28
	.003"-.0065"	3	12	2	8
	<.003"	0	0	1	4
		18	72	11	44
Corundum	.01"-.02"	5	20	1	4
	.0065"-.01"	16	64	7	28
	.003"-.0065"	5	20	8	32
	<.003"	0	0	6	24
		26	104	22	88
Spinel	.003"-.0065"	2	8	0	0
	<.003"	1	4	0	0
		3	12	0	0
Staurolite	.01"-.02"	60	240	2	8
	.0065"-.01"	>100	> 400	13	52
	.003"-.0065"	>200	> 800	12	48
	<.003"	1	4	3	12
		>361	>1,444	30	120

Results of Tests for Refractory-Type Particles in Heavy Minerals Separated from Carolina Silica Co. Sand (Pilot Processed by N.C.S.U. Mineralogical Labs.), Treated with HCl & HNO_3, and Dispersed in Regular Glass Melt

Sample No. RML-5, Lab. No. TP-74-360

Mineral Constituent	Least Dimension of Particle	Quantities of Particles Observed Dispersed in Glass Melt: Particles in Melt Heated for 1-Hour @ 2400° F — From 25.0 lb. Sample	Particles in Melt Heated for 1-Hour @ 2400° F — Per 100 lbs.	Particles in Melt Heated for an Additional 1-Hour @ 2600°F — From 25.0 lb. Sample	Particles in Melt Heated for an Additional 1-Hour @ 2600°F — Per 100 lbs.
Chromite	.003"-.0065"	3	12	1	4
	<.003"	2	8	0	0
		5	20	1	4
Zircon	.01"-.02"	1	4	0	0
	.0065"-.01"	4	16	0	0
	.003"-.0065"	33	132	3	12
	<.003"	68	272	2	8
		106	424	5	20
Kyanite	.01"-.02"	114	456	4	16
	.0065"-.01"	~400	~1,600	13	52
	.003"-.0065"	~500	~2,000	19	76
	<.003"	~150	~ 600	1	4
		~1,164	~4,656	37	148
Andalusite	.01"-.02"	9	36	0	0
	.0065"-.01"	11	44	0	0
	.003"-.0065"	0	0	0	0
	<.003"	0	0	0	0
		20	80	0	0
Corundum	.01"-.02"	3	12	1	4
	.0065"-.01"	2	8	0	0
	.003"-.0065"	2	8	0	0
	<.003"	1	4	0	0
		8	32	1	4
Staurolite	.01"-.02"	8	32	0	0
	.0065"-.01"	20	80	0	0
	.003"-.0065"	26	104	0	0
	<.003"	0	0	0	0
		54	216	0	0

Results of Tests for Refractory-Type Particles in Heavy Minerals Separated from Carolina Silica Co. Sand (Pilot Processed by N.C.S.U. Mineralogical Labs.), Treated with HCl & HNO_3, and Dispersed in Regular Glass Melt

Sample No. RML-6, Lab. No. TP-74-361

Mineral Constituent	Least Dimension of Particle	Quantities of Particles Observed Dispersed in Glass Melt: Particles in Melt Heated for 1-Hour @ 2400° F — From 25.0 lb. Sample	Particles in Melt Heated for 1-Hour @ 2400° F — Per 100 lbs.	Particles in Melt Heated for an Additional 1-Hour @ 2600°F — From 25.0 lb. Sample	Particles in Melt Heated for an Additional 1-Hour @ 2600°F — Per 100 lbs.
Chromite	.0065"-.01"	1	4	0	0
	.003"-.0065"	2	8	1	4
	<.003"	4	16	1	4
		7	28	2	8
Zircon	.0065"-.01"	4	16	0	0
	.003"-.0065"	26	104	1	4
	<.003"	82	328	1	4
		112	448	2	8
Kyanite	.02"-.03"	3	12	0	0
	.01"-.02"	54	216	8	32
	.0065"-.01"	92	368	11	44
	.003"-.0065"	>300	>1,200	4	16
	<.003"	82	328	0	0
		>531	>2,124	23	92
Andalusite	.01"-.02"	4	16	0	0
	.0065"-.01"	9	36	0	0
	.003"-.0065"	15	60	0	0
	<.003"	0	0	0	0
		28	112	0	0
Corundum	.01"-.02"	1	4	0	0
	.0065"-.01"	4	16	0	0
	.003"-.0065"	5	20	0	0
	<.003"	0	0	0	0
		10	40	0	0
Staurolite	.01"-.02"	7	28	0	0
	.0065"-.01"	16	64	0	0
	.003"-.0065"	27	108	0	0
	<.003"	6	24	0	0
		56	224	0	0

Results of Tests for Refractory-Type Particles in Heavy Minerals Separated from Carolina Silica Co. Sand (Pilot Processed by N.C.S.U. Mineralogical Labs.), Treated with HCl & HNO_3, and Dispersed in Regular Glass Melt

Sample No. RML-8, Lab. No. TP-74-362

Mineral Constituent	Least Dimension of Particle	Quantities of Particles Observed Dispersed in Glass Melt: Particles in Melt Heated for 1-Hour @ 2400°F — From 25.0 lb. Sample	Particles in Melt Heated for 1-Hour @ 2400°F — Per 100 lbs.	Particles in Melt Heated for an Additional 1-Hour @ 2600°F — From 25.0 lb Sample	Particles in Melt Heated for an Additional 1-Hour @ 2600°F — Per 100 lbs.
Chromite	.0065"-.01"	1	4	0	0
	.003"-.0065"	0	0	1	4
	<.003"	3	12	0	0
		4	16	1	4
Zircon	.01"-.02"	3	12	0	0
	.0065"-.01"	8	32	4	16
	.003"-.0065"	~100	~400	0	0
	<.003"	~300	~1,200	5	20
		~411	~1,644	9	36
Kyanite	.02"-.03"	21	84	0	0
	.01"-.02"	89	356	1	4
	.0065"-.01"	>300	>1,200	15	60
	.003"-.0065"	>200	>800	9	36
	<.003"	65	260	0	0
		>675	>2,700	25	100
Andalusite	.01"-.02"	10	40	0	0
	.0065"-.01"	0	0	0	0
	.003"-.0065"	3	12	0	0
	<.003"	0	0	0	0
		13	52	0	0
Staurolite	.01"-.02"	6	24	0	0
	.0065"-.01"	11	44	1	4
	.003"-.0065"	21	84	1	4
	<.003"	0	0	0	0
		38	152	2	8

Results of Tests for Refractory-Type Particles in Heavy Minerals Separated from Carolina Silica Co. Sand (Pilot Processed by N.C.S.U. Mineralogical Labs.), Treated with HCl & HNO_3, and Dispersed in Regular Glass Melt

Sample No. RML-9, Lab. No. TP-74-363

Mineral Constituent	Least Dimension of Particle	Quantities of Particles Observed Dispersed in Glass Melt: Particles in Melt Heated for 1-Hour @ 2400°F — From 25.0 lb. Sample	Particles in Melt Heated for 1-Hour @ 2400°F — Per 100 lbs.	Particles in Melt Heated for an Additional 1-Hour @ 2600°F — From 25.0 lb. Sample	Particles in Melt Heated for an Additional 1-Hour @ 2600°F — Per 100 lbs.
Chromite	---	None	---	---	---
Zircon	.01"-.02"	1	4	0	0
	.0065"-.01"	26	104	2	8
	.003"-.0065"	~1,000	~4,000	4	16
	<.003"	~ 150	~ 600	10	40
		~1,177	~4,708	16	64
Kyanite	.02"-.03"	10	40	0	0
	.01"-.02"	42	168	0	0
	.0065"-.01"	> 100	> 400	3	12
	.003"-.0065"	> 500	>2,000	7	28
	<.003"	33	132	1	4
		> 685	>2,740	11	44
Andalusite	.01"-.02"	3	12	0	0
	.0065"-.01"	9	36	1	4
	.003"-.0065"	9	36	0	0
	<.003"	0	0	0	0
		21	84	1	4
Corundum	.01"-.02"	1	4	0	0
	.0065"-.01"	0	0	1	4
	.003"-.0065"	0	0	0	0
	<.003"	0	0	0	0
		1	4	1	4
Staurolite	.01"-.02"	1	4	0	0
	.0065"-.01"	48	192	1	4
	.003"-.0065"	> 100	> 400	1	4
	<.003"	0	0	1	4
		> 149	> 596	3	12
Al-Refractory	.03"-.04"	1	4	0	0
	.01"-.02"	0	0	1	4
		1	4	1	4

Summary of Heavy Minerals Test Results

(100 lb. Sample Basis)

Lab No.	Sample Marking	Total Heavy Minerals Weight	Total Heavy Minerals Percent	HCl & HNO_3 Treated Heavy Minerals Weight	HCl & HNO_3 Treated Heavy Minerals Percent	Plus 70 Mesh Refractory Weight
TP-74-358	RML-3	2.736 gms.	.0060	1.628 gms.	.0036	.5898 gm.
TP-74-359	RML-4	3.536 gms.	.0078	2.652 gms.	.0058	Not determined.
TP-74-360	RML-5	3.844 gms.	.0085	2.260 gms.	.0050	Not determined.
TP-74-361	RML-6	2.620 gms.	.0058	1.324 gms.	.0029	.2828 gm.
TP-74-362	RML-8	5.644 gms.	.0124	3.704 gms.	.0082	.5540 gm.
TP-74-363	RML-9	9.068 gms.	.0200	7.068 gms.	.0156	.7520 gm.
				MODEL SAND SPEC.:		.200 gm. Maximum

Summary of Amounts of Refractory-Type Particles Surviving 1-Hour @ 2400°F Plus 1-Hour @2600°F in Regular Glass Melts

Mineral Constituent	Least Dimension of Particle	Number of Refractory-Type Particles Surviving Glass Melt Tests (100-lb. Sample Basis) RML-3 TP-74-358	RML-4 TP-74-359	RML-5 TP-74-360	RML-6 TP-74-361	RML-8 TP-74-362	RML-9 TP-74-363
Chromite	.01"-.02"	---	4	---	---	---	---
	.0065".01"	104	8	---	---	---	---
	.003"-.0065"	56	4	4	4	4	---
	<.003"	32	12	---	4	---	---
		192	28	4	8	4	
Zircon	.0065"-.01"	8	12	---	---	16	8
	.003"-.0065"	8	172	12	4	---	16
	<.003"	---	>300	8	4	20	40
		16	484	20	8	36	64
Kyanite	.02"-.03"	---	12	---	---	---	---
	.01"-.02"	40	92	16	32	4	---
	.0065"-.01"	36	196	52	44	60	12
	.003"-.0065"	100	>400	76	16	36	28
	<.003"	---	20	4	---	---	4
		176	>720	148	92	100	44
Andalusite	.01"-.02"	---	4	---	---	---	---
	.0065"-.01"	12	28	---	---	---	4
	.003"-.0065"	---	8	---	---	---	---
	<.003"	---	4	---	---	---	---
		12	44				4
Corundum	.01"-.02"	4	4	4	---	---	---
	.0065"-.01"	---	28	---	---	---	4
	.003"-.0065"	---	32	---	---	---	---
	<.003"	---	24	---	---	---	---
		4	88	4			4
Staurolite	.01"-.02"	---	8	---	---	---	---
	.0065"-.01"	---	52	---	---	4	4
	.003"-.0065"	---	48	---	---	4	4
	<.003"	---	12	---	---	---	4
			120			8	12
Al-Refractory	.01"-.02"	---	---	---	---	---	4

E.H.Cunningham/lh