DEPARTMENT OF MINES AND RESOURCES

BUREAU OF MINES

CANADA

Ottawa, February 17, 1947.

REPORT

of the

MINERAL DRESSING AND METALLURGICAL LABORATORIES.

Investigation No. 2093.

Beneficiation of a Siliceous Sand from Barrington Bay, Nova Scotia.

Note:
This report relates essentially to the samples as received. It shall not, nor any correspondence connected therewith, be used in part or in full as publicity or advertising matter for the sale of shares in any promotion.

(Copy No. 16.)

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R E P O R T

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Beneficiation of a Siliceous Sand from Barrington Bay, Nova Scotia.

Shipment:

Two hundred and seventy pounds of sand was received on February 20, 1946, from Mr. J. P. Messervey, Mining Engineer, Nova Scotia Department of Mines, Halifax, Nova Scotia. In addition some 10 or 15 pounds of this sand had previously been received from Mr. L. H. Cole of the Mineral Resources Division for preliminary work. (Mr. Cole in addition arranged for the shipment of the 270 pounds.)

Location of the Property:

The sample was taken from the sand dunes at Subine Beach on the north side of Barrington Beach at Barrington Bay, Shelburne county, Nova Scotia.

Sampling, Analysis and Screen Test:

After a thorough mixing of the shipment a head sample was cut out by standard methods which analysed as follows:

		Per Cent
SiO_2	-	81.02
Fe_2O_3	-	0.90
Al_2O_3	-	11.68
CaO	-	2.28
MgO	-	0.44
L.O.I.	-	0.74

A screen test on this material reported as follows:

Mesh		Weight, Per Cent
+35	-	4.0
-35 +48	-	17.6
-48 +65	-	43.8
-65+100	-	31.2
-100+150	-	2.6
-150+200	-	0.3
-200	-	0.5
Total	-	100.0

Microscopic Examination:

Under the binocular microscope the silica particles appear to be sub-angular to angular, clear to translucent; a few are milky. Some of the longer grains have incipient fractures which might break down when used for sand-blasting. Very few splintery fragments were observed, the grains being mostly flat with equal dimensions. The impurities noted in this binocular examination were: hornblende, tourmaline, ilmenite, rutile, kaolin, muscovite, phlogopite, biotite, sericite, lepidolite, and a few pieces of feldspar. A large number of the quartz particles were pitted with a dark material which was thought to be hornblende. Iron-stained quartz was also prevalent.

In the examination of the individual sizes obtained from the screen test, the plus 35 mesh material showed the

(Microscopic Examination, cont'd) -

largest amount of impurities, which were mostly in the form of biotite mica and hornblende. The smaller sizes of minus 100 mesh showed much weathered or iron-stained quartz, muscovite mica and organic material. An approximate count of the percentage of impurities in the different sizes reported as follows:

Mesh		Impurities, Per Cent
+35	-	6
-35 +48	-	3
-48 +65	-	5
-65+100	-	2
-100+150	-	6
-150+200	-	8
-200	-	10

These impurities were in addition to the "middling material" or those grains of quartz which contained numerous small pitted intrusions. There was also considerable milky white or opaque grains showing. This was probably a form of aluminium silicate mineral which has been rounded by wind and water action. They probably were originally a variety of feldspar.

Purpose of Investigation:

It was required to ascertain whether it was possible, by ore dressing procedure, to obtain a beneficiated sand from this material which would be suitable for glass making.

Investigative Work:

The test work on this 270-pound shipment included washing, tabling, flotation, high-power magnetic concentration, roasting with salt and washing, and, finally, an acid treatment with H_2SO_4. A combination of washing and magnetic separation gave a product assaying 0.17 per cent Fe_2O_3, 83.46 per per cent SiO_2, 10.74 per cent Al_2O_3, 0.034 per cent TiO_2.

(Investigative Work, cont'd) -

When this product was subjected to sulphuric acid treatment and again washed, a final product assaying 0.06 Fe_2O_3, 83.88 per cent SiO_2, 10.76 per cent Al_2O_3, 0.025 per cent TiO_2 was obtained.

Numerous flotation tests were conducted on the washed sands with mediocre results. On the smaller samples, obtained from Mr. L. H. Cole, a number of small 50-gram flotation tests were made with a view to studying different reagents to indicate the possibilities of flotation treatment. Details of the test work follow:

- Screening and Washing -

After the screen test and microscopic examination, as previously given, had indicated that a large percentage of the impurities were in the plus 35 mesh and minus 100 mesh material, it was decided to eliminate the plus 35 mesh in all preliminary procedure prior to the test work. This entails a primary loss of some 4 per cent of plus 35 mesh material. In some of the tests the minus 100 mesh size was also screened out, which was a further loss of 3.4 per cent.

Test No. 1.

A portion of the sample was passed through a 35-mesh screen and the plus 35 mesh material was collected and assayed.

The minus 100 mesh was also screened out and the remaining minus 35 plus 100 mesh portions weighed and assayed.

Results:

Product	Weight, per cent	Assays, per cent			Distribution of Fe_2O_3, per cent
		Fe_2O_3	Al_2O_3	SiO_2	
Feed	100.0	0.90	11.68	81.02	100.0
+35 mesh	5.2	1.25	--	--	8.8
-35+100 mesh	93.3	0.69	9.38	81.76	87.3
-100 mesh	1.5	1.91	--	--	3.9

(Investigative Work, cont'd) -

Test No. 2.

In this test a portion of the sand was treated as in Test No. 1 and the remaining minus 35 plus 100 mesh portion washed. This washing procedure was done in a 6,000 c.c. enameled pail with a spout to drain off the overflow material. 2,000 grams of the screened sand (-35+100) was placed in the pail and a stream of water introduced. The pulp was then stirred continuously for 10 minutes and the overflow portion retained. This test was run in duplicate and the following results obtained:

Product	Weight, per cent	Assay, Fe_2O_3, per cent
Feed	100.0	--
Overflow	5.2	--
Washed sand	94.8	0.63
Feed	100.0	0.90
Overflow	6.6	--
Washed sand	93.4	0.64

A microscopic examination of the washed sand showed that perhaps two-thirds of the mica had been washed out. The sand still contained numerous grains of hornblende and some grains of ilmenite and limonite. "Middling products" were still numerous, the quartz grains containing pitted enclosures of hornblende and biotite.

The overflow material contained a large proportion of biotite mica; some fine iron-stained quartz and also a little black organic matter were visible.

Test No. 3 (A, B, C, D)

- Flotation Concentration -

A portion of the sand was screened and washed as in Tests Nos. 1 and 2. The pulp was then divided into four parts, A, B, C and D, and subjected to flotation concentration in a "Fagergren" machine as follows: In Tests A and B,

(Investigative Work, cont'd) -

anionic reagents were used with an alkaline pH, and in Tests C and D cathionic reagents were tried with an acid pH. Results were as follows:

Test	Product	Weight, per cent	Assays, per cent SiO_2	Al_2O_3	Fe_2O_3
A	Flot. tailing	88.0	79.71	12.13	0.75
B	" "	88.9	80.48	11.58	0.68
C	" "	97.8	81.41	10.96	0.75
D	" "	96.1	81.84	10.67	0.54

- Reagents -

Test	Conditioned with	Activated with	Floated with	pH
A	--	1 lb./ton $PbNO_3$	0.6 lb./ton sodium oleate	7.8
B	--	1 lb./ton $PbNO_3$	0.4 lb./ton sodium oleate); 0.1 lb./ton cresylic acid)	7.8
C	3 lb./ton H_2SO_4	2 lb./ton $CuSO_4$	1.5 lb./ton DP 243	4.0
D	2 lb./ton HF	1.7 lb./ton $CuSO_4$	1.1 lb./ton DP 243	4.2

The times used in conditioning and activating were 5 minutes and in flotation, 7 minutes. A microscopic examination of the concentrates showed that in A and B the predominent mineral was quartz, with some biotite mica and hornblende. In Tests C and D, biotite mica comprised 15 to 20 per cent of the concentrate with some hornblende and middling products.

Test No. 4 (A, B, C, D)

- Magnetic Concentration -

In this test the screened and washed material was dried and passed through a Dings high-intensity magnetic machine at a voltage of 100 and amperage of 2.0. The results were as follows:

(Continued on next page)

(Investigative Work, cont'd) -

Test:	Product	Weight, per cent	Assay, Fe_2O_3, per cent
A	Non-mag. tailing	90.8	0.21
B	" "	83.5	0.20
C	" "	87.5	0.22
D	" "	90.0	0.23

A microscopic examination of the magnetic concentrates showed biotite and muscovite mica, hornblende, tourmaline and a great many "middling products," i.e., quartz grains which were pitted with mica or hornblende.

Test No. 5 (A, B, C, D)

- Flotation of Non-magnetic Tailing from Test No. 4 -

In this test, flotation concentration of these non-magnetic tailings was performed as follows: A portion of the tailings were pulped with distilled water in a Fagergren cell and in Tests A, B and C activated with 0.2 pound per ton $PbNO_3$ and floated with reagents as noted. In Test D the pulp was conditioned with 2.0 pounds per ton H_2SO_4, activated with 1.5 pounds per ton $CuSO_4$, and floated. The results were as follows:

Test No.:	Product	Weight, per cent	Flotation Reagent, lb./ton	Assay, Fe_2O_3, per cent	pH
5A	Flot. tailing	93.9	AMAC 1120	0.17	7.3
B	" "	94.0	Sodium oleate	0.19	7.8
C	" "	98.0	Rosin amine D	0.16	7.2
D	" "	97.9	AMAC 1120	0.15	4.5

A microscopic examination of the flotation concentrates showed little mica, the flotation consisting in the main of hornblende, tourmaline, and "middling products".

(Investigative Work, cont'd) -

Test No. 6 (A and B)

- Table Concentration -

Two portions of the sand were screened and washed as in previous tests and the remaining products passed over a laboratory-size Wilfley table.

Results:

Test No.	Product	Weight, per cent	Assay, Fe_2O_3, per cent
6A	Table tailing	92.3	0.50
6B	" "	93.4	0.51

There was no clean-cut line of demarcation in the table concentration. A microscopic examination of these table concentrates showed considerable biotite mica with some hornblende and middling products.

Test No. 7.

- Salt Roasting and Washing -

A portion of the screened and washed sand was mixed with 5 per cent of C.P. NaCl and heated for one hour at a temperature of 1900° F. The calcine was then cooled, dried, weighed, washed, and sampled.

Results:

The washed roasted product assayed 0.65 per cent Fe_2O_3 with a loss of 5.0 per cent. The microscopic examination showed a great deal of iron-stained quartz with a somewhat lesser amount of mica and hornblende. A large number of middling products also were visible.

Test No. 8.

100 pounds of sand was taken and the plus 35 mesh material screened out. The remaining portion was washed in a small spiral classifier at a feed rate of 100 pounds per hour. The resulting underflow, or washed sand, was divided

(Investigative Work, cont'd) -

into two portions. Table concentration followed by magnetic concentration of the table tailing was practised on one lot, while on the other portion of the washed sand a series of flotation tests was performed. In addition, a portion of the non-magnetic tailings was used for further flotation tests and another portion for a series of sulphuric acid heating and washing treatments.

Results:

- Screening and Washing -

Product	Weight, per cent	Assay, per cent Fe_2O_3	Distribution, per cent Fe_2O_3
Feed	100.0	0.90	100.0
+35 mesh	3.7	1.25	4.6
Class. overflow	1.8	2.01	3.6
Class. cleanout	5.7	5.00	28.5
Class. underflow	88.8	0.60	53.3

- Table Concentration of Washed Sand Underflow -

Product	Weight, per cent	Assay, per cent Fe_2O_3	Distribution, per cent Fe_2O_3
Feed	100.0	0.60	100.0
Table conc.	4.5	2.30	22.0
Table tail'	95.5	0.49	78.0

As before, there was no clean-cut line in this tabling test. The microscope showed hornblende, ilmenite, tourmaline and very little mica in the table concentrate. Numerous "middling" grains were also visible.

- Magnetic Concentration -

Another portion of the washed sand was passed through the Ding magnetic high-intensity machine at 100 volts, 2 amperes.

(Continued on next page)

(Investigative Work, cont'd) -

Results:

Product	Weight, per cent	Assays, per cent Fe_2O_3	TiO_2	Al_2O_3	SiO_2
Feed	100.0	0.60	--	--	--
Mag. conc.	11.1	--	--	--	--
Non-mag. conc.	88.9	0.17	0.034	10.74	83.46

This test shows 74.8 per cent of the Fe_2O_3 was removed in the magnetic concentrate. Four portions of this non-magnetic tailing were subjected to flotation concentration. Results were as follows:

Product Feed		Weight, per cent	Assay, Fe_2O_3, per cent
Flot. tailing	A	95.2	0.15
" "	B	94.9	0.17
" "	C	98.0	0.16
" "	D	97.9	0.16

- Reagents Used -

Flot. No.	Conditioner	Activator	Collector	pH
A	--	$PbNO_3$(.2 lb.)	AMAC 1120(.2 lb.)	7.2
B	--	$PbNO_3$(.2 lb.)	Rosin amine D(.2 lb.)	7.2
C	H_2SO_4(2 lb.)	$CuSO_4$(1.4 lb.)	Rosin amine D(.2 lb.)	4.4
D	H_2SO_4(a lb.)	$CuSO_4$(1.4 lb.)	AMAC 1120(.2 lb.)	4.2

Microscopic examination of these flotation tailings showed that numerous "middling" products contained the bulk of the iron impurities. There was also a considerable amount of milky and opaque silicates.

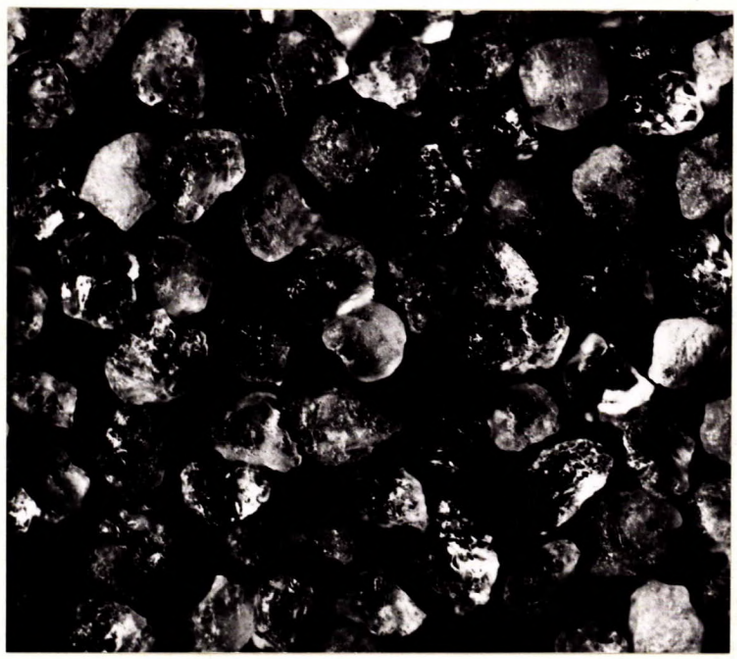
A screen test on the non-magnetic tailing reported as follows:

Mesh		Weight, per cent
+35	-	Nil.
-35+48	-	16.8
-48+65	-	59.1
-65+100	-	23.9
-100	-	0.2

(Investigative Work, cont'd) -

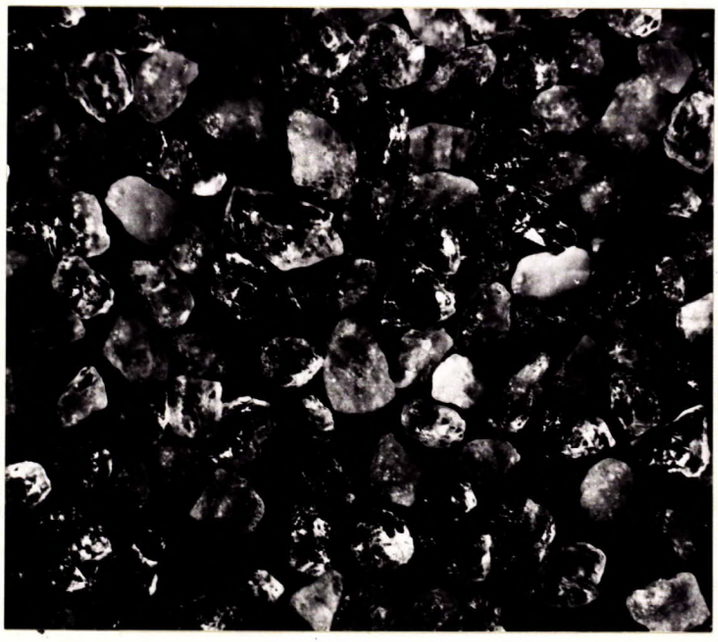
In order to further illustrate the shape of these different particle sizes, the following pictures are shown:

Figure 1.



-35+48 MESH.

Figure 2.



-48+65 MESH.

(Investigative Work, cont'd) -

Figure 3.

-65+100 MESH.

It should be noted that the darker portions of the particles are due to shadows.

- Flotation Concentration -

Other portions of the screened, washed sand were subjected to flotation concentration, with the following results:

Flot.	Product	Weight, per cent	Assay, Fe_2O_3, per cent
A	Flot. tailing	61.4	0.45
B	" "	94.0	0.47
C	" "	98.8	0.56
D	" "	98.5	0.52
E	" "	87.2	0.57
F	" "	96.7	0.50
G	" "	98.3	0.54

(Investigative Work, cont'd) -

Reagents (lb./ton)

Flot.	Conditioner	Activator	Collector	pH
A	2 lb. H_2SO_4	--	3.0 lb. 801; 2 lb. fuel oil	4.0
B	--	0.4 lb. $PbNO_3$	0.15 lb. AMAC 1120	5.8
C	1 lb. H_2SO_4	1.0 lb. $CuSO_4$	0.25 lb. " "	4.4
D	1 lb. "	2.0 lb. "	0.20 lb. " "	4.4
E	--	0.6 lb. $PbNO_3$	0.30 lb. sodium oleate	6.9
F	--	0.6 lb. "	0.30 lb. rosin amine D	6.7
G	1 lb. H_2SO_4	1.2 lb. $CuSO_4$	0.30 lb. rosin amine D	4.4

Conditioning time (H_2SO_4), 5 minutes; activating time ($PbNO_3$ or $CuSO_4$), 10 minutes; flotation time, 7 minutes.

Under the microscope the flotation concentrates showed black particles of biotite mica, iron-stained quartz, and a little hornblende and tourmaline. The flotation tailing still contained numerous "middling" grains, a few pieces of iron-stained quartz, and some hornblende and tourmaline. There was little free mica remaining in this product.

- Sulphuric Acid Heat Treatment and Wash -

As a final test, portions of the non-metallic tailings from the Dings machine, assaying 0.17 per cent Fe_2O_3, were treated with sulphuric acid and washed. Different quantities of the tailings were mixed with 5 per cent or 10 per cent solutions of H_2SO_4 and heated at 80° C. for a 10-hour period in an "Anco" constant temperature oven. The resulting product was then cooled and transferred to the washing apparatus (previously described) and washed for a 10-minute period. This final product was dried and assayed for Fe_2O_3.

Results:

Product	Weight, grams	c.c. of 10% H_2SO_4 Added	Fe_2O_3 in Final Washed Product, per cent
A	200	25	0.060
B	500	50	0.040
C	750	75	0.080
		c.c. of 5% H_2SO_4	
D	200	50	0.046
E	500	100	0.074
F	750	150	0.062

(Investigative Work, cont'd) -

The above results are approximately consistent. The amount of H_2SO_4 added is roughly equivalent to 25 pounds per ton of sand and the cost at 1 cent per pound would be 25 cents per ton. A microscopic examination of the treated product showed the Fe impurities to consist mostly of iron stains on the quartz grains. Some milky or opaque crystals of aluminium silicate minerals are to be seen and also a few grains of clear quartz attached to the milky or opaque mineral.

SUMMARY AND CONCLUSIONS:

The test work on the shipment showed that some 96.6 per cent of the sand screened minus 28 plus 100 mesh. It was found advisable to discard the plus 35 mesh size, leaving 92.6 per cent of the sand available for further beneficiation. Washing, tabling, high-intensity magnetic separation, flotation concentration, and, finally, acid heat treatment methods were all tried out. The head sample assayed 0.90 per cent Fe_2O_3. By washing in a spiral classifier an iron content of 0.60 per cent Fe_2O_3 was obtained. This washed sand was dried and passed through a Dings high-intensity magnetic machine and a non-magnetic tailing assaying 0.17 per cent Fe_2O_3, 83.46 per cent SiO_2, 10.74 per cent Al_2O_3, 0.034 per cent TiO_2 resulted. This tailing was heated with sulphuric acid and washed, giving a final product assaying 0.06 per cent Fe_2O_3, 83.88 per cent SiO_2, 10.76 per cent Al_2O_3, 0.025 per cent TiO_2.

Table concentration was not effective on this sand, tabling tests on the primary washed product not giving any distinct line of demarcation and only lowering the iron content from 0.60 per cent Fe_2O_3 to 0.49 per cent Fe_2O_3.

(Continued on next page)

(Summary and Conclusions, cont'd) -

A large number of flotation tests were conducted on the primary washed sand. These tests gave mediocre results. The cause of this failure was apparently the larger number of "middling products" in the sand. These products contained numerous small pitted cavities which were filled with foreign material (mica, hornblende, etc.) and would not respond to flotation.

The best results were obtained by the use of the Dings high-intensity magnetic machine. Using a voltage of 100 and amperage of 2.0, this machine was able to pull off a large amount of "middlings product" in addition to the free mica and hornblende which had remained after the washing treatment. There still was left, however, a considerable number of particles of quartz and aluminium silicates which were slightly iron stained and contained minute intrusions of biotite mica and hornblende.

The sulphuric acid heat treatment and following wash still left some of these impurities in the final product as evidenced by the assay of 0.06 per cent Fe_2O_3.

From the above summary of the results obtained, it is apparent that the present methods of beneficiation as employed in this investigation, coupled with the fact of the large number of "middling particles" in the sand, would not be sufficient to produce a final product which would be acceptable for the manufacture of glass. It may be that in the next few years flotation reagents will be developed that will improve beneficiation.

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