

GLASS-SAND POTENTIAL OF THE PINEHURST
FORMATION IN RICHMOND COUNTY, NORTH
CAROLINA: PROCESSING AND MINERAL
CHARACTERIZATION

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INTRODUCTION/SUMMARY:

The North Carolina Geological Survey requested process options to make a glass sand product from a silica sample out of Richmond County, North Carolina. The underlying sedimentary strata of the Coastal Plain covering the crystalline rocks of the Piedmont, characterize the Richmond County site. The primary formations represented in outcropping are the Pinehurst and the Middendorf (Figure 1, Appendix), both potential sources for glass-quality quartz sand. Six drill cores (Figure 2, Appendix) were taken from east-west and north-south directions on the site and were used in the analysis of glass sand (Chem-Nuclear Systems, 1993).

Sedimentary silica sand is a common source for commercial silica, mainly used for glass containers in the United States. The sand must be free of mica, organic matter, iron, titanium, trace elements, or

characterization of the Richmond County area for a low-level radioactive waste disposal facility. The drill holes were selected based on their crosscutting relationships with loose sand and not chosen based on horizontal distances from each other (Chem-Nuclear Systems, 1993).

Table 1. Drill Hole Data

Drill Hole	Depth Below Surface (meters)	Number of Samples	Samples Used In Composite	Entire Depth of Sample (meters)
R42	9.14	15	6	1.07-2.90
R63	18.29	37	11	0.30-6.10
R83	8.53	43	29	0.00-5.94
R41	9.14	24	16	0.30-5.94
R9	9.14	45	7	0.30-4.27
R18	10.97	53	48	0.00-10.36

Based on observation, the composite sample from hole R42 was composed of fine to very coarse-grained sand with a light tan color. This sample contained very coarse quartz with a slight orange stain.

The sample from hole R63 was composed of medium to very fine-grained light tan sand. The quartz was cleaner in this sample compared to the more prominent iron stains on other samples. The whiter color might have been an indication of kaolin present in the sample.

The R83 sample contained fine to medium-grained tan sand. Mica was observed in the sample.

The R41 sample contained very fine to medium-grained, white to light tan sand with a slight orange stain. R41's color was lighter or with less stain than most of the other samples. Mica was observed in the composite sample.

The R9 sample consisted of fine to coarse-grained medium to dark tan sand with small amounts of orange and dark brown stain.

This sample had a deeper brown color compared to the other samples. More organic material was in the sample.

The sample from hole R18 consisted of fine to coarse-grained, orange to red stained sand. A deep brown stain was also present. From 0.00 to 5.33 meters the sand appeared to be a light orange to light brown color. From 5.33 to 10.36 meters the color became a deep red corresponding to depth. Due to the apparent contrast between these two depths, tests were done utilizing the first 5.33 meters (a total of twenty-three samples combined) and the next 5.03 meters (a total of twenty-five samples combined). Tests were done on the combined 10.36 meters also.

Sample Preparation

The composite sample from each hole was ground in a laboratory rod mill with three rods for 30 seconds to one minute to loosen large clumps. This milling action thoroughly mixed the sample. Then the sample was split into 400-to 700-gram portions to ensure equal distribution of soil components and to prepare them for subsequent testing.

Scrubbing

For five minutes, 500-gram samples were scrubbed at 70% solids at 1200 RPM. The pulp pH was maintained at less than three with sulfuric acid (10 wt %). After scrubbing, the samples were deslimed on a 200-mesh screen. The deslimed products were further evaluated using percent solids, pH, and varied scrubbing time, whereas the slimes were flocculated, settled, filtered, dried, and weighed (Tables 2, 4).

Sizing/Screening

The deslimed scrubbed products were filtered and dried at 150°C overnight. Each sample was sieved on a Ro Tap using US Standard 30-, 40-, 60-, 100-, 140-, 200-, and -200-mesh. These size fractions were weighed and bagged after ten minutes of screening, and the weight distribution was calculated.

Magnetic Separation

Size fractions from 40-to 140-mesh were combined and used for magnetic separation. All samples went through three passes on the magnetic separator (Permroll) at 150 RPM. Tests 6719-2, -4, and -6 combined 40-, 60-, and 100-m size fractions whereas tests 6719-3, -5, and -7 combined 40-, 60-, 100-, and 140-m sizes for the magnetic separation. The size fractions combined were determined by the overall weight percent for each mesh size. Only the largest weight percentages were used for magnetic separation.

Heavy Liquid Separation

Heavy liquid was utilized to separate the heavy minerals as sink product, and the glass sand as the float product. Heavy liquid (tetrabromoethane) was used on 150 to 160 grams of the original composite samples to separate minerals with specific gravities greater than 2.96. All sinks were settled, filtered, and then cleaned with isopropyl alcohol. Floats were filtered and washed with alcohol. The floats and sinks were left to dry in an oven overnight, and then weighed and bagged.

Flotation Process Evaluation

The flotation process consisted of conditioning the material at 60-70% solids with a petroleum sulfonate collector for approximately five minutes at a pH <3 maintained with sulfuric acid. The conditioned pulp was adjusted to 25% solids and the iron mineral impurities were floated as the float product. The machine discharge was dewatered and conditioned at approximately 40% solids for one minute with an amine and a frother at a maintained pH less than three. The conditioned pulp was adjusted to 25% solids and the mica floated as the float product. The machine discharge was then dewatered and conditioned at around 40% solids for one minute with an amine, HF, and frother at a maintained pH less than three. The conditioned pulp was adjusted to 25% solids and the feldspar impurities floated as the float product leaving the machine discharge as the final glass sand

product. All flotation products were filtered, dried, weighed, and assayed (Table 3).

Chemical Analysis

The standard chemical analysis for glass sand by XRF (Fe_2O_3 , Al_2O_3 , CaO , MgO , TiO_2 , K_2O , SiO_2 , ZrO , Na_2O , P_2O_5) and LOI (Loss on Ignition) was completed by The Mineral Lab, Inc. for six samples. Their methods are as follows:

A representative portion of each sample was ground to approximately -400 mesh in a tungsten carbide swing mill (to avoid Fe contamination) and then analyzed by their standard "XRF-powder" procedure for 31 major, minor and trace elements. The relative precision/accuracy for this procedure is approximately five to ten percent for major and minor elements, and approximately ten to 15 percent for trace elements at levels greater than twice the detection limit in samples of average geologic composition. A replicate sample and a standard reference material ("SY3" a CANMET standard rock) were analyzed with the samples to demonstrate analytical reproducibility for samples and analytical accuracy for a geologic standard, respectively. The accepted "known" values for the quality control standard are listed with the XRF results. W and Co were omitted from the list of results because these elements were contaminants contributed by the tungsten carbide mill.

A representative portion of each sample was dried for two hours at 105°C , cooled to room temperature in a desiccator, and weighed to determine the weight percent moisture loss or MOI. Each dried and weighed sample was returned to the furnace, heated at $1,000^\circ\text{C}$ for four hours, cooled to room temperature in a desiccator and re-weighed to determine the weight percent Loss on Ignition or LOI. A replicate sample was tested for quality control (Mineral Lab Results, 2000).

Mineral Identification-Qualitative Analysis

Flotation products were further analyzed by powder x-ray diffraction. (Feldspar floats did not yield a substantial amount suitable for XRD analysis, and no feldspar was visible under the

microscope.) A small amount of sample was ground in a mortar and pestle. The powder was placed on a mount and evenly dispersed with one to two drops of ethanol. Once dry, the samples were scanned for identification of minerals at $0.2^\circ 2\theta/\text{sec}$, with a generator tension of 45 kV and 40 mA.

RESULTS:

Table 2. Attrition Scrubbing Tests – Individual Drill Holes*

Test Number	6719-1	6719-2	6719-3	6719-4	6719-5	6719-6	6719-7
Sample ID	R83	R83	R41	R18	R63	R9	R42
<i>Cumulative % Retained:</i>							
US 40 mesh	28.0	26.5	41.0	33.6	37.2	45.3	52.9
US 60 mesh	58.1	56.9	69.2	62.9	57.2	73.9	75.2
US 100 mesh	84.8	84.7	91.0	88.7	76.3	92.6	91.4
US 140 mesh	91.4	91.4	95.7	94.4	81.1	96.2	95.7
Passing 140 mesh	8.6	8.6	4.3	5.6	3.7	3.7	4.3
Scrub Yield Wt. %	80.1	80.3	73.8	77.3	62.4	71.9	63.0
Final Non-magnetic Yield Wt. %	72.7	70.1	70.2	69.5	59.3	66.9	59.8
<i>Product Chemical Analysis Wt %:</i>							
Fe ₂ O ₃		0.02	0.02	0.01	0.02	0.04	0.02
Al ₂ O ₃		0.15	0.13	0.14	0.13	0.34	0.16
MnO		0.01	0.01	0.01	0.01	0.01	0.01
TiO ₂		0.01	0.01	0.01	0.01	0.02	0.01
K ₂ O		0.01	0.01	0.01	0.01	0.01	0.01
Na ₂ O		0.05	0.05	0.05	0.05	0.05	0.05
MgO		0.05	0.05	0.05	0.05	0.05	0.05
CaO		0.01	0.01	0.01	0.01	0.01	0.01
P ₂ O ₅		0.05	0.05	0.05	0.05	0.05	0.05
SiO ₂		99.6	99.8	98.7	99.1	97.2	98.8
LOI		0.01	0.1	0.07	0.09	0.1	0.1

*scrub % solids 70%; scrub reagent H₂SO₄

Table 3. Flotation Tests*

Test Number	6719-8	6719-9	6719-10	6719-11	6719-12
Sample ID	R83	R18 17.5-34ft	R18 0- 34ft	R41	R18 0- 17.5ft
<i>Cum. % Retained:</i>					
US 40 mesh	30.7	28.1	33.2	39.4	37.8
US 60 mesh	60.7	57.3	62.1	68.1	66.2
US 100 mesh	86.4	85.5	88.2	91.0	90.9
US 140 mesh	93.3	93.5	94.6	96.3	96.3
Passing US 140 mesh	6.7	6.6	5.4	3.7	3.8
Float Yield, Wt. %	80.1	81.0	78.0	74.9	75.5
Final Non-magnetic Yield Wt. %	78.3	78.9	76.7	73.8	74.1

*scrub % solids 70%; scrub reagent H₂SO₄; iron, mica, and feldspar conditions 70% solids

Table 4. Attrition Scrubbing Tests – Composites

Test Number	6719-13	6719-14	6719-15	6719-16	6719-17	6719-18
Scrub % Solids	70	70	65	65	70	70
Scrub Reagent	NaOH	H ₂ SO ₄	H ₂ SO ₄	NaOH	None	H ₂ SO ₄
Scrub Time (min.)	5.0	5.0	5.0	5.0	5.0	3.0
<i>Cumulative % Retained:</i>						
US 40 mesh	28.5	33.2	33.0	34.2	31.0	31.3
US 60 mesh	56.7	62.4	62.1	62.8	61.1	60.4
US 100 mesh	84.4	87.8	87.7	87.7	87.2	86.7
US 140 mesh	92.0	94.2	94.1	93.8	94.1	93.4
Passing 140 mesh	8.0	5.9	5.9	6.2	5.9	6.5
Scrub Yield Wt. %	79.1	78.5	78.3	76.9	80.9	78.6
Final Non-magnetic Yield Wt. %	75.2	76.9	76.5	74.6	78.8	76.5
<i>Product Chemical Analysis Wt. %</i>						
Fe ₂ O ₃	0.02	0.03	0.03	0.02	0.04	0.03

Al ₂ O ₃	0.16	0.22	0.23	0.14	0.22	0.21
MnO	0.01	0.01	0.01	0.01	0.01	0.01
TiO ₂	0.02	0.03	0.02	0.02	0.02	0.02
K ₂ O	0.01	0.01	0.01	0.01	0.01	0.01
Na ₂ O	0.05	0.05	0.05	0.05	0.05	0.05
MgO	0.05	0.05	0.05	0.05	0.05	0.05
CaO	0.01	0.01	0.01	0.01	0.01	0.01
P ₂ O ₅	0.05	0.05	0.05	0.05	0.05	0.05
SiO ₂	98.0	96.5	97.0	97.2	98.4	98.2
LOI	0.08	0.07	0.09	0.1	0.08	0.1

Most of the raw material ranged between 60 to 100 mesh, although significant portions of sand were in the +40 and +140 size fractions. The specifications of glass sand usually require the largest amount of material to be between 40 and 140 mesh (Personal Communication, J. Schlanz, 2001).

The yield of sand after scrubbing ranged from 62.4 to 80.3%. The overall yield of the non-magnetic product ranged from 59.3 to 72.7% (Table 2). Flotation results ranged from 73.8 to 78.9% (Table 3). The overall yield of non-magnetic product in the composite scrubbed samples ranged from 74.6 to 78.8% (Table 4).

For the composite samples, a natural pH scrub done on test number 6719-17 showed satisfactory results, having a final non-mag yield weight percentage of 78.8 and a fairly low Fe₂O₃ and Al₂O₃ content. Test number 6719-16 had the lowest non-mag yield weight percentage but a lower Fe and Al content (Table 4).

All of the heavy liquid separations yielded floats being nearly 99% of the weight. All sinks were less than one gram.

Chemical analysis results revealed that all weight percentages for major elements were low (Table 4). For all samples, iron (Fe₂O₃) was below 0.04%, and at least one sample, 6719-3, had less than 0.01 percent. All percentages of magnesium (MgO) were below the detection limit of 0.05%. Percent aluminum in the samples ranged from 0.34% (6719-5) to 0.13% (6719-2,-4). Titanium (TiO₂)

of a stain on the quartz. Immediately after the initial scrubbing and desliming, much of the iron stain was eliminated from the quartz product. The remainder of iron and other magnetic minerals would be removed during magnetic separation.

The further scrubbing done on composite samples showed a higher final non-mag yield weight percentage than the scrubbed individual drill hole samples. This is a good indication that even though individual drill holes may contain an initial inferior quartz quality, the composite sample taken as a whole had an improved quality.

The flotation tests were completed only on individual drill hole samples and not on composite samples. These results showed high final non-mag yield weight percentages, although no chemical analysis was finalized on these samples to affirm low percentages of unwanted minerals. Flotation experiments performed on a composite sample probably would show an increase in quartz quality. However, flotation requires more chemicals, equipment, and labor, so the cost would be higher to produce this glass grade quartz using flotation.

For tests that have higher amounts of +40 mesh material, crushing could be an option to increase product value. If excessive amounts of -140 mesh are present, screening the fine material could also be beneficial. Excessive fines are undesirable and could cause small persistent flaws in the glass that is being produced (Murphy, 1960). Further tests done on the -140 mesh could be considered for other uses so that the waste material during processing would be minimal.

percentages ranged from 0.03 in 6719-14 to less than the detection limit of 0.01 in 6719-2, -3. In the composite samples, titanium and aluminum contents were slightly higher but were still well within requirement specifications. Trace elements in all of the samples also were very low and measured in parts-per-million.

The products of the iron flotations analyzed by x-ray diffraction yielded primarily quartz. After separation of the quartz from the bulk of the sample under a microscope, the x-ray results indicated the presence of schorl (tourmaline), zircon, and rutile. When viewed under the microscope, most of the grain sizes had a dark reddish-brown color. This could have been an indication that fractions of the quartz were stained with goethite and hematite, or the color could have indicated the minerals mentioned above, schorl, zircon, and rutile. The mica flotations also contained a fair amount of quartz (40%), but trace minerals such as kaolinite and muscovite were also detected. Feldspar was undetectable in the sample as indicated by XRD analysis and by sight observation through a microscope (Personal Communication, B.J. Allen, 2000).

CONCLUSION:

XRF analysis showed that each sample was glass grade quality according to The Industrial Minerals HandyBook standards. This specific type of glass sand could be used for the following: glass grade, ceramic grade, foundry grade silica, filtration sand, and possibly sodium silicate feedstock and silica flour. All of the beneficiation methods were successful in producing glass sand that met requirement specifications. The chemical analysis focused on impurities in the samples and the percent difference of silica, rather than the actual silica content. All of the major and minor element requirements for glass grade sand were met, including the most crucial: aluminum, iron, titanium, potassium, chromium, and zircon standards.

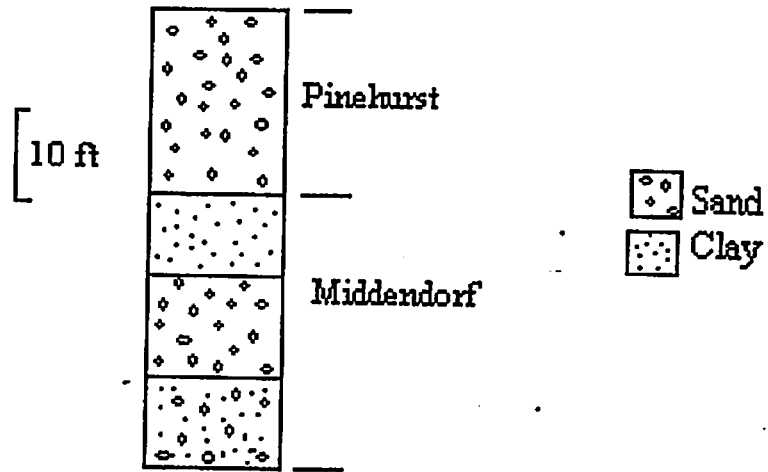
The sand can be evaluated at two levels, one using a scrubbing technique along with magnetic separation, and the other using flotation and magnetic separation. The scrubbing or flotation processes would eliminate much of the iron because it is in the form

References

- Brady, G.S. and Clauser H.R., 1977, *Materials Handbook*, 11th Edition: McGraw-Hill Book Co., NY 773 p.
- Chem-Nuclear Systems, Inc., 1993, *Site Characterization Report for the Richmond County Potentially Suitable Site: Raleigh, NC*, 68 p.
- Harben, P.W., 1999, *Silica and Quartz in The Industrial Minerals HandyBook*, 3rd Edition: Industrial Minerals Information Ltd., Surrey, UK, p.183-190.
- Mills, N., 1983, *Glass Raw Materials, Industrial Minerals and Rocks*, 5th Edition: The American Institute of Mining, Metallurgical, and Petroleum Engineers, Inc., NY, p. 339-347.
- Murphy, T.D., 1960, *Silica Sand and Pebble in Industrial Minerals and Rocks*, 3rd Edition: The American Institute of Mining, Metallurgical, and Petroleum Engineers, Inc., NY, p. 763-772.
- The Mineral Lab Inc., October 13, 2000, *Methods and Results*, 2 p.

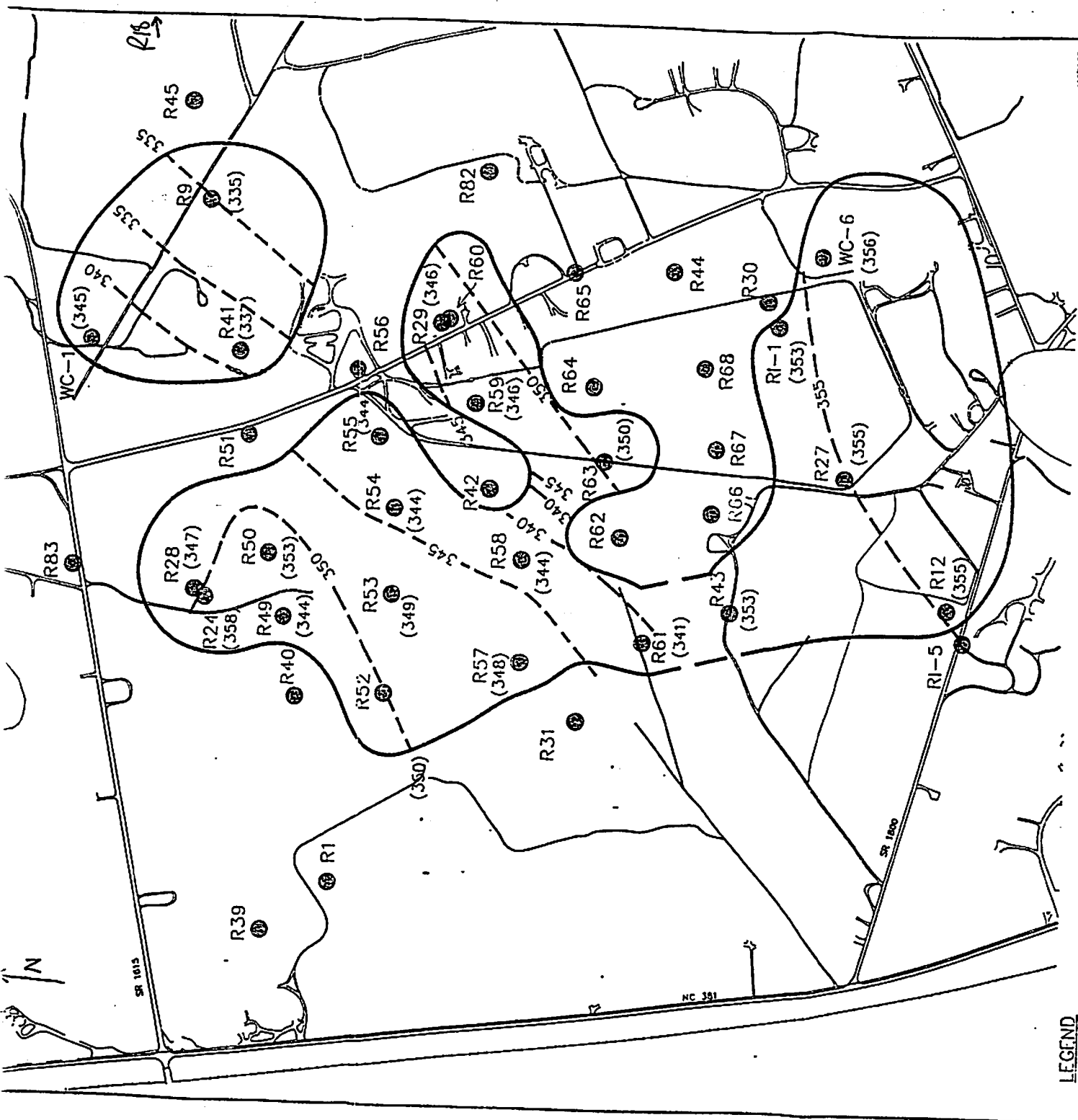
APPENDIX

Figure 1.



Typical Drill Hole Cross-Section of the Richmond County Site (R63)

Figure 2.



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STRUCTURE CONTOURS AT BASE OF SHALLOW CLAY ZONES IN AREA OF PROPOSED DISPOSAL CELL CONSTRUCTION



LEGEND
— BOUNDARY OF CLAY ZONE
- - - 350 ELEVATION CONTOUR LINE