

HIGH-SILICA SAND POTENTIALITIES OF  
THE OHIO RIVER FORMATION

*by*  
ARTHUR P. PINSAK

Indiana Department of Conservation  
GEOLOGICAL SURVEY  
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# HIGH-SILICA SAND POTENTIALITIES OF THE OHIO RIVER FORMATION

BY ARTHUR P. PINSAK

## ABSTRACT

Laboratory tests indicate that sand from the Ohio River formation, a deposit of probable Tertiary age, can be treated so as to meet the requirements of high-silica sand by means of simple and inexpensive beneficiation methods that will effectively remove most of the iron and alumina from the sand.

Skewness curves and histograms indicate that the sand is well sorted and has an average median grain size of 0.26 millimeter. Petrographic studies show that only stable minerals are present in the sand. Although heavy minerals constitute an average of only 0.425 percent of the sand, the relative amount of heavy minerals is uniform in all sand samples. Composition of the sand suggests a granitic source, and texture of the heavy minerals indicates at least four cycles of deposition of the sand. This study suggests that the Ohio River formation was deposited during Pliocene time in a fluvial environment.

Differences in color of the various units of the formation are due to secondary oxidation of the contained iron. X-ray diffraction analysis established the fact that iron is present in the formation principally as ferruginous clay. An acid or base leach proved to be the most effective treatment for removing this type of contamination.

All beneficiation methods which were tested increase the purity of the sand above minimum high-silica specifications. The best results were obtained, however, with a water-washed sand placed in an 18 N solution of sulfuric acid for a period of 40 minutes at a temperature of 140° C. This leaching method produced a sand that meets specifications for first quality glass sand.

## INTRODUCTION

### PURPOSE OF STUDY

All high-silica sand used at present in Indiana is shipped from other states. During the past several years, however, an interest has arisen in possible sources of high-silica sand within the state.

The Ohio River formation, a sand deposit that is generally considered to be Tertiary in age, is one of several geologic formations that were used for manufacturing glass toward the end of the nineteenth century. Like most other sands and sandstones in Indiana and elsewhere, sand from the Ohio River formation contains too many impurities in its natural state to meet present-day specifications for glass sand and some other high-silica uses. Impurities must be removed by some sort of treatment.

The purpose of this study, therefore, was to determine the physical, chemical, and mineralogical properties of sand from the Ohio River formation and to establish experimental methods for removing its impurities.

#### COLLECTING LOCALITIES AND LABORATORY METHODS

Samples were collected from exposures of the Ohio River formation at the junction of Floyd, Washington, and Clark Counties, at sites in southeastern Harrison County, and at a pit at the junction of Hardin and Meade Counties near the town of Tip Top, Ky. Mechanical, petrographic, and X-ray powder diffraction analyses of the sand were made. Various methods of beneficiation were used in treating the samples, and products resulting from beneficiation were analyzed with a spectrograph to determine the effectiveness of the methods which were used.

#### FIELD WORK

Field work was done during the summer of 1952. Samples were collected from abandoned pits and from natural exposures. One week during August 1952 was spent with Dr. William J. Wayne in studying areal distribution of the formation in Indiana and in examining the pit near Tip Top, Ky. Locations from which samples were taken are shown in figure 1.

#### PREVIOUS WORK

The Ohio River formation has received the attention of many workers but little detailed work has been done. G. H. Ashley (1902, p. 68-70) named the formation and noted its occurrence in Harrison County and at scattered locations in Washington County, Ind. He apparently considered the deposits to be of marine origin. C. A. Malott (1922, p. 134) described occurrences of the formation and considered it to be a beach deposit. E. F. Burchard (1907, p. 366), C. H. Richardson (1920, p. 93), Charles Butts in a letter to Frank Leverett (1929, p. 11-12), Frank Leverett (1929, p. 8-12), and L. L. Ray, A. P. Butler, Jr., and C. S. Denny (1947) also have written about the occurrence of the formation. Murray and Patton (1953, p. 24-25, 29) included some samples and analyses used in this report.

#### PREVIOUS USE OF THE SAND

Sand from the formation was used by the DePauw Glass Works of New Albany, Ind., late in the nineteenth century. The sand also

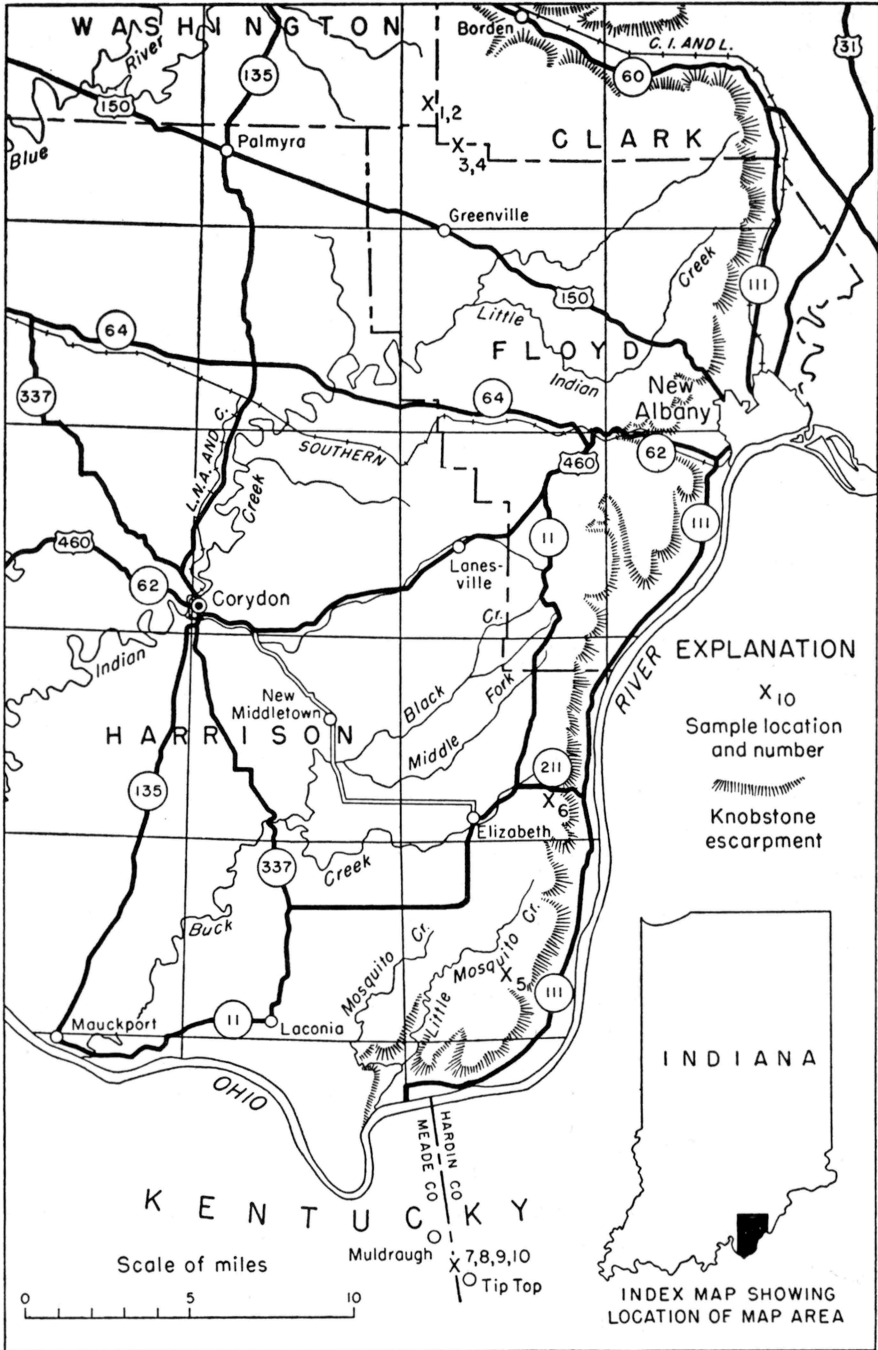


Figure 1.—Map showing collecting localities of samples of sand and facilities for transporting sand.

has been used locally as a mortar sand. The pit near Tip Top, Ky., shipped high-silica sand until 1941, when the United States Government acquired the property.

#### ACKNOWLEDGMENTS

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#### USES AND REQUIREMENTS FOR HIGH-SILICA SAND

Deposits suitable for production of high-silica sand should be friable or unconsolidated in order to eliminate costly methods of extracting and crushing. The deposits also should be extensive and easily accessible.

Glass sands are found in many geologic formations. The Oriskany sandstone of West Virginia and Pennsylvania is widely used, and Tertiary sands of New Jersey are used to some extent. By far the best known and most extensively used glass sand in the United States is the St. Peter sandstone of Chazyan age. This sand, known commercially as the Ottawa sand, is mined and quarried in Illinois and Missouri.

Good grades of sand are available on the market, but glass producers, in an attempt to lower costs, are interested in a source of sand near their plants. As mentioned above, all high-silica sand now used in Indiana is imported from other states.

High-silica sand is used extensively as a constituent in manufacturing glass, as engine sand, paving sand, and filter sand, as an abrasive in scouring powders, sand paper, sandblasting, and gang saws, and, after suitable bonding clay has been added, as foundry sand.

Glass manufacturers apply exceedingly stringent specifications to sand. Impurities must be at a minimum, as iron causes color

and opacity, clay causes cloudy glass, and magnesia, lime, and alumina are highly refractory and increase the time required to melt the sand. Grain size must be uniform in order to insure an even melt. Coarse grains tend to melt slowly, and fines tend to blow out of the batch and thus cause a loss in output per ton of sand.

The American Ceramic Society and the National Bureau of Standards have set up the following specifications for chemical composition and grain size of glass sands (see tables 1 and 2); specifications may vary slightly among the individual producers (Ries, 1949, p. 972-973).

Table 1.—*Specifications for chemical composition of glass sands*

Quality	SiO <sub>2</sub> minimum (pct.)	Al <sub>2</sub> O <sub>3</sub> maximum (pct.)	Fe <sub>2</sub> O <sub>3</sub> maximum (pct.)	CaO + MgO maximum (pct.)
First quality, optical glass.....	99.8	0.1	0.020	0.1
Second quality, flint-glass containers and tableware .....	98.5	0.5	0.035	0.2
Third quality, flint glass.....	95.0	4.0	0.035	0.5
Fourth quality, sheet glass, rolled and polished plate .....	98.5	0.5	0.060	0.5
Fifth quality, sheet glass, rolled and polished plate .....	95.0	4.0	0.060	0.5
Sixth quality, green glass containers and window glass .....	98.0	0.5	0.300	0.5
Seventh quality, green glass.....	95.0	4.0	0.300	0.5
Eighth quality, amber glass.....	98.0	0.5	1.000	0.5
Ninth quality, amber glass.....	95.0	4.0	1.000	0.5

Table 2.—*Sieve specifications for grading of glass sands*

Passing sieve	Retained on sieve	Percentage
No. 20 .....		100
No. 20 .....	No. 40	40-60
No. 40 .....	No. 60	30-40
No. 60 .....	No. 100	10-20
No. 100 .....		0-5

## PHYSIOGRAPHY

The area in which the Ohio River formation occurs in Indiana is in the extreme south-central part of the state in eastern Harrison County and western Floyd County, on the west edge of Clark County, and at scattered points in Washington County. The area is on the Highland Rim Plateau of the Interior Low Plateaus and lies within the Interior Plains region of North America (Fennemman, 1928, p. 307-309). The formation lies along the eastern boundary of the physiographic province known as the Mitchell

plain (Beede, 1911, p. 95), and its present areal extent is largely controlled by dissection of the crest of the Knobstone escarpment (Malott, 1922, p. 90), which lies directly to the east.

The Mitchell plain, developed on the St. Louis and Ste. Genevieve limestones, is a southwestward-sloping limestone plain with well-developed karst topography. The slope of the surface is about 20 feet per mile. The beds dip to the west at a rate of about 35 feet per mile and thus expose successively older strata to the east. Karst topography is best developed on the St. Louis limestone, which crops out at the east edge of the plain. The eastern boundary of the Mitchell plain is difficult to determine because this plain merges with the Norman upland (Malott, 1922, p. 92) to the east. The Norman upland is a rolling, maturely dissected upland area composed of lower Mississippian clastic rocks. Divides capped by limestone characteristic of the Mitchell plain extend far into the upland area, and deep valleys possessing the characteristics of the Norman upland extend for great distances into the limestone plain. The western boundary of the Norman upland is marked by the Knobstone escarpment.

The Knobstone escarpment in Harrison, Floyd, and Clark Counties and in southeastern Washington County forms a divide between steeply descending streams that flow eastward to the Ohio River and deeply entrenched streams that meander westward across the Mitchell plain to the Blue River or Ohio River on the west side of Harrison County.

## STRATIGRAPHY

The Ohio River formation is composed largely of unconsolidated but well-compacted sand. The sand ranges in color from red to white and exhibits an irregular, haphazardly arranged system of crossbedding. Exposures reach a maximum thickness of 64 feet at Tip Top, Ky. A section 80 feet thick was reported southeast of Buena Vista in Harrison County (Malott, 1922, p. 134), but a section measuring 80 feet could not be found.

The sand rests unconformably on the Salem limestone in Clark County and on the St. Louis limestone in Harrison County. The elevation of the sand above sea level ranges from 700 feet at Tip Top, Ky., to 920 feet in southeastern Washington County.

Vertical breccia zones composed of fragments of white sand in a red sand matrix and fractures within the sand indicate that post-depositional slumping of the sand into solution cavities took place.

The sand is present as remnants which cap limestone divides and which are separated by deep V-shaped valleys. Although the sand is found on the higher ridges and knobs of the area, limestone ridges showing no evidence of sand are present east of the exposures at elevations exceeding those of the sand. The sand deposits appear to have been thicker originally, but some of the sand has been removed by erosion subsequent to deposition.

Most of the outcrops consist of two units below the overburden: an upper red zone and a lower white zone. At Tip Top, Ky., however, four units are represented: an upper red, upper medial white, lower medial tan, and a lower white unit. The medial tan unit has been observed at some localities in Indiana, but the lowest white unit is not exposed at the localities which were sampled and may not be present in Indiana. Although the sand is well compacted, it crumbles when it is removed from the face of an exposure. The samples from Indiana represent units ranging from 9.2 to 16.8 feet in thickness.

## DESCRIPTIONS OF SAMPLE LOCALITIES

### MILLER SAND PIT

An old sand pit known as the Miller pit is in the center of the NE $\frac{1}{4}$  sec. 18, T. 1 S., R. 5 E., 2 miles southeast of Martinsburg and 4 miles south of Pekin, Washington County.

The following section, located on the westward-facing wall and 50 feet from the south end of the pit, was measured and sampled.

## HIGH-SILICA SAND POTENTIALITIES

*Stratigraphic section measured and sampled in Miller sand pit*

		<i>Feet</i>	<i>Sample</i>
Overburden	Soil: brownish-gray in upper half; becomes fine clay with reddish-brown mottling in lower part..	7.0	
Overburden	Clay: yellow to bright red with a few white streaks; arenaceous; shows no crossbedding...	1.0	
11	Sand: interlaminated red and yellow bands approximately one-eighth inch thick; some fine crossbedding .....	0.7	}
10	Sand: light-tan with few dark laminae.....	0.6	
9	Sand lens: reddish-violet .....	0.6	
8	Sand: evenly interbedded tan and grayish-orange to reddish-tan bands; dark bands darkest at top and fade into grayish-orange sand at base..	1.9	
7	Sand: reddish-brown lens; lateral extent limited.	0.2	
6	Sand: grayish-orange and tan; shows some crossbedding; individual bands dark at top and light at bottom; brown lens 3.4 to 3.6 feet from top of unit .....	3.7	
5	Sand: tan at top; grades to white at bottom....	0.4	
4	Sand: white; few scattered grayish-orange beds; better consolidated than upper part of section..	2.5	
3	Sand: alternating tan and white bands, three-quarters inch thick; contains some crossbedding	0.8	
2	Sand: reddish-tan lens .....	0.2	
1	Sand: white; has some grayish-orange partings; well compacted .....	2.0	
	(Water was encountered at this point.)		
	Total thickness of measured section.....	21.6	
	Total thickness of sampled section.....	13.6	



The following section, also located on the westward-facing wall, is 100 feet north of the section shown above. The base of this section is 1.8 feet higher than the base of the first section.

*Stratigraphic section measured and sampled in Miller sand pit*

		<i>Feet</i>	<i>Sample</i>
11	Sand: alternating tan and yellow bands; dark band at bottom one-eighth inch thick.....	0.8	}
10	Sand: white .....	0.2	
9	Sand: alternating brown and reddish-purple bands	0.6	
8	Sand: grayish-orange .....	0.6	
7	Sand: predominantly yellow with a few white and brown laminae .....	4.4	
6	Sand: grayish-orange; a few white bands.....	1.1	
5	Sand: yellow with white bands at base; some crossbedding .....	1.6	
4	Sand: white with yellow laminae one-eighth inch wide and 0.3 foot apart.....	1.2	
3	Sand: white; very compact .....	1.4	
2	Sand: brown lens 1 foot long.....	0.2	
1	Sand: white; two grayish-orange zones 2 inches wide; very compact .....	2.7	
	Total thickness of measured section.....	14.8	
	Total thickness of sampled section.....	14.8	

The overburden has been stripped from the northern part of the Miller sand pit at the place where the second section is located. The pit consists of a vertical wall facing west and trending north-south for approximately 250 feet. The entire wall shows a very irregular system of crossbedding.

A small pond at the south end of the pit stands at ground-water level. The first section was sampled to the level of the pond, which lies approximately 920 feet above sea level. The sand terminates at the south end of the pit, and here the ground is slightly dissected, as it is for 300 feet or more to the west.

The topography of the area surrounding the pit is flat to gently rolling. A drilling program would be necessary in order to determine the extent and thickness of the sand to the north and east, the two directions in which it most probably persists.

A water supply is lacking in the immediate vicinity; a large pond is not present and the only water courses consist of small, intermittent streams scattered throughout the area. The pit is 3.8 miles north of Greenville, which is 18 miles west of Louisville, Ky., on U. S. Highway 150. The closest rail connection is the Chi-

cago, Indianapolis and Louisville Railroad, which passes through Pekin and Borden.

#### JORRIS SAND PIT

An old sand pit, known locally as the Jorris pit, is in the SE $\frac{1}{4}$  NW $\frac{1}{4}$  sec. 20, T. 1 S., R. 4 E. It is 2.5 miles north of Greenville and 3 miles southeast of Martinsburg, Clark County.

The following section, located at the extreme south end of the pit and facing eastward, was measured and sampled.

*Stratigraphic section measured and sampled in Jorris sand pit*

		<i>Feet</i>	<i>Sample</i>
Overburden	Clay: gray to tan; reddish-brown mottling.....	4.6	
11	Sand: light-red with a few scattered dark-red bands; very compact, almost friable sandstone..	1.3	}
10	Sandstone: reddish-brown; persistent laterally; very limonitic .....	0.02	
9	Sand: yellow and white banded; yellow predominant at top, white at bottom; very compact.....	2.48	
8	Sand: white; pale-red and yellow zones 3.0 to 3.5 inches thick; compact .....	1.2	
7	Sand: reddish-orange; thin white streak.....	0.5	
6	Sand: white; a few tan to pale grayish-orange thin bands; fairly compact.....	1.2	
5	Sand: white to grayish-orange; pale-red alternating bands 0.25 to 0.3 inches wide becoming narrower toward bottom .....	1.8	
4	Sand: grayish-orange and tan, interbedded; slightly less compact than units above; a light-brown band at 14.4 feet .....	2.9	
3	Sand: white; pale grayish-orange interbedding...	4.2	
2	Sand: light-brown .....	0.5	
1	Sand: white .....	0.4	
	(Water was encountered at this point.)		
	Total thickness of measured section.....	21.1	
	Total thickness of sampled section.....	16.5	

The following section, also facing eastward, was measured and sampled 48 feet north of the preceding section. The base of this sampled section lies 0.9 feet higher than the base of the first section measured and sampled in the Jorris sand pit.

*Stratigraphic section measured and sampled in Jorris sand pit*

		<i>Feet</i>	<i>Sample</i>
	Red sand and overburden stripped from this section .....	11.0	
8	Sand: white with tan crossbedding.....	0.5	}
7	Sand: orange pink; red banding; some red clay concretions .....	0.9	
6	Sand: white; scattered grayish-orange to tan crossbedding; fairly compact .....	5.0	
5	Sand: yellowish-brown with white streaks.....	0.6	
4	Sand: white with pale grayish-orange interbedding	3.8	
3	Sand: tan and white, interbedded; some lenses..	1.5	
2	Sand: light reddish-brown .....	0.3	
1	Sand: white; a few scattered tan bands.....	0.5	
	Total thickness of measured section.....	24.1	
	Total thickness of sampled section.....	13.1	

The outcrop is in an old pit which has not been extensively worked in recent years. The pit consists of an eastward-facing wall, which is approximately 150 feet long. The wall is dissected by deep gullies, especially at the north end where the overburden has been stripped. A small spring flows out of the bottom of the wall.

The base of the outcrop lies approximately 920 to 930 feet above sea level. The sand apparently rests unconformably on the Salem limestone, as an outcrop was seen in a gully 720 feet south-east of the pit at an elevation between 900 and 910 feet.

The land slopes to the east, is well dissected in this direction, and is dotted with sinkholes. Sand occurs in a deep gully which lies 450 feet north of the pit. To the west, south, and northwest, however, the land is gently rolling, and drilling would be necessary to determine the lateral extent of the sand.

A large water supply is not available in the vicinity; only small intermittent streams flow across the area. The closest railroad is the Chicago, Indianapolis and Louisville Railroad, which passes through Pekin 5.5 miles to the north and through Borden 5.2 road miles to the north-northeast.

BUENA VISTA

An exposure is located at the junction of secs. 21, 22, 27, and 28, T. 5 S., R. 5 E., in Harrison County. The outcrop lies directly south of a county road that runs along a ridge 1½ miles south of Buena Vista.

*Stratigraphic section measured and sampled at an exposure south of Buena Vista*

		Feet	Sample
Overburden	Clay: brownish-gray at top; tan grading into red at bottom; very sandy; more compact at base than at top .....	7.0	
9	Sand: tan to reddish-brown; crossbedded; slump fractures filled with red clay; red clay stringers caused by rotted plant roots.....	1.6	}
8	Sand: white, pink, and yellow zones; some red and tan banding; crossbedded; very well compacted	4.6	
7	Sand: white .....	0.6	
6	Sand: buff-colored; light at center becoming very brown at bottom; base contains small clay balls	0.6	
5	Sand: tan; grading to grayish-orange at base; crossbedded .....	0.9	
4	Sand: brown; irregular crossbedding; less compacted than unit 5 .....	0.4	
3	Sand: white, tan, and yellow crossbedding.....	1.4	
2	Sand: red and white, banded.....	0.2	
1	Sand: white; pale grayish-orange and pink banding .....	2.7	
	Total thickness of measured section.....	20.0	
	Total thickness of sampled section.....	13.0	

The base of the sampled section is not the base of the formation. An attempt was made to take an auger sample at the base of the sampled section, but the sand is too well compacted to bore into it with an ordinary soil auger. A 2-foot section consisting of interbedded tan and white sand was sampled but was not considered representative enough to be included in sample no. 5.

Sample no. 5 was taken from the deepest gully in the area. Only 10.1 feet could be sampled at any one point. It was necessary to move 41 feet to the south along a horizontal marker bed to sample the lowest 2.9 feet. The sand is well exposed at a lower elevation a short distance farther south. However, the exposures could not be correlated with certainty, and as the outcrop appears to be dipping southward, it was thought best to sample only 13 feet in order to avoid resampling stratigraphic equivalents.

The terrain around the sampled section is dissected by a series of gullies trending southward to the headwaters of Little Mosquito Creek; it resembles badland topography. The outcrop is bounded on the east by Bearwallow Ridge. East of the ridge, sand could not be found; only clay and gravel were present. The section apparently rests unconformably on the St. Louis limestone. A St. Louis

outcrop was observed in a deep gully 1,000 feet north of the county road at an elevation between 740 and 760 feet and 80 to 100 feet below the top of the sampled section.

A large water supply, which would be needed to work the deposit, is not available in the immediate vicinity. The closest sizable body of water is the Ohio River, which lies 2.2 miles east and 440 feet lower than the sand. The streams in the area are all intermittent.

This location is about 17 miles south of New Albany by way of the Ohio River or Indiana Highway 111. New Albany is the closest railroad connection. The other railroad in the area is the Louisville, New Albany, and Corydon Railroad, which passes through Corydon, 22 miles to the northwest.

SAND BANK CORNER

An exposure is located at the east edge of the SE $\frac{1}{4}$ , SW $\frac{1}{4}$  sec. 26, T. 4 S., R. 5 E., 2.3 miles east-northeast of Elizabeth, Harrison County.

*Stratigraphic section measured and sampled at Sand Bank Corner*

		<i>Feet</i>	<i>Sample</i>
Overburden	Clay: gray to red.....	7.0	
7	Sand: white; yellow banding near bottom; compact .....	0.9	}
6	Sand: grayish-orange to yellow at top grading to tan at bottom; haphazardly scattered dark-brown fracture planes and root shoots.....	2.5	
5	Sand: tan to yellow; scattered red bands; very compact .....	2.1	
4	Sand: grayish-orange; very well compacted.....	0.7	
3	Sand: white; buff to brown mottling caused by leaching of the overburden .....	1.1	
2	Sand: white; less compact than unit 3.....	1.1	
1	Sand: brownish-yellow; some pink laminae.....	0.8	
	Total thickness of measured section.....	16.2	
	Total thickness of sampled section.....	9.2	

Overburden 2 to 3 feet thick had to be removed in order to obtain an exposure of the section. Units 1, 2, and 3 were taken from a bank 100 feet northeast of the upper part of the section. The top of unit 3 was leveled from the bottom of a similar unit.

The sand is well compacted at the top of the section and is looser and easier to remove toward the bottom. The whole section contains intricate crossbedding such as one might expect to find in a fluvial deposit.

The bank lies on the west side of a deeply eroded former stream channel which opens onto the Ohio River flood plain 1 mile to the east at Stewart's Landing. An old sand pit on the A. Lawson property lies 1,500 feet to the south at the head of this same gully.

The old pit reported to be at this location lies 400 feet east of the bank alongside a county road. The old pit has been covered during recent years but does not appear to have ever been deep.

The area lying west of the sampled section is flat to gently rolling except for a few gullies caused by stream erosion. There is no reason to suspect that the Ohio River formation does not underlie at least part of the area. A drilling program would be needed to determine the areal extent of the sand.

The base of the sampled section is approximately 800 feet above sea level. A limestone quarry is 0.4 mile north-northeast on the north side of a county road. St. Louis limestone crops out at the top of the quarry at an elevation of 790 feet. The Ohio River formation apparently lies unconformably on top of the St. Louis limestone at Sand Bank Corner.

The closest large water source is the Ohio River, which is 1.4 miles east and approximately 420 feet lower than the deposit.

The outcrop lies 1.4 miles west of the Ohio River, 2.4 road miles east of Elizabeth, 9.1 road miles southeast of New Middleton, 15.6 road miles southeast of Corydon, and 12 miles south of New Albany on Indiana Highway 111. All the roads except those within 2 miles of the outcrop are macadam-surfaced.

The closest railroad except those at New Albany is the Louisville, New Albany, and Corydon Railroad at Corydon.

#### TIP TOP, KENTUCKY

A pit is located on the line between Meade and Hardin Counties, Ky., and is 0.6 mile west-northwest of Tip Top, Ky., and 1.1 miles south of Muldraugh, Ky., on the Fort Knox Military Reservation.

*Stratigraphic section measured and sampled near Tip Top, Ky.*

		Feet	Sample
Overburden	Soil: brown, arenaceous at base.....	7.0	
16	Sand: white to grayish-orange; fairly well compacted; tan and red crossbedding; many irregular dark-red bands that are probably rotted plant roots and fracture fillings.....	5.3	}
15	Sand: red; loosely compacted .....	0.8	
14	Sand: grayish-orange; pink, tan, and yellow bands and laminae; compact .....	0.8	
13	Sand: buff to pale-red; tan, red, and white crossbedding becoming lighter toward bottom.....	5.0	
12	Sand: grayish-orange; pink and tan laminae and crossbedding; one 2-inch black band at 30° truncated at top .....	1.8	
11	Sand: reddish-orange; fine grayish-orange and red laminae .....	0.7	
10	Sand: grayish-orange to buff; thin tan, pink, and red bands and laminae; fairly well compacted..	3.7	
9	Sand: black; soft; probably manganese oxide stain	0.1	
8	Sand: white; a few irregularly spaced tan and grayish-orange bands and laminae; fairly well compacted .....	6.0	
7	Sand: gray and tan banded; some bands almost black (mottled); very soft .....	1.1	
6	Sand: white to grayish-orange; irregular tan bands; some crossbedded laminae.....	1.9	
5	Sand: gray with black spots; tan to grayish-orange interbedding; very soft.....	1.3	
4	Sand: grayish-orange with pinkish-tan crossbedding .....	2.5	
3	Sand: grayish-orange to bright yellow; very irregularly bedded alternating grayish-orange and yellow units; some crossbedded laminae toward bottom; each bed reaches a maximum darkness at bottom .....	7.0	}
2	Sand: white; fine grayish-orange to tan laminae irregularly spaced through unit; fairly well compacted .....	5.0	}
1	Covered interval down to a very shallow pond in the west-central part of the pit; around pond is scattered float consisting of botryoidal, concentrically laminated chalcedony, weathered chert nodules, limestone fragments, and chips of brachiopods and fenestellid bryozoans.....	14.0	

Total thickness of measured section..... 64.0

Total thickness of sampled section..... 43.0

The pit extends about 1,500 feet in an easterly-westerly direction, and the width is about half the length. The section is thickest at the northwest end of the pit and thins in all other directions.

In general, the surface of the area is rolling and is dotted with sinkholes. A small abandoned pit was discovered north of the pit from which the sample was taken. The two pits are separated by the Illinois Central Railroad. The tracks apparently had stopped operations in a northerly direction, and a highway had confined operations to the south.

An undulating clayey shale interval crops out around the walls of the pit. The shale is green, red, and tan and reaches a maximum thickness of 1.5 feet. This unit dips below the bottom of the pit on the north face in the area which was sampled; it appears again farther east on the north face of the pit on the east side of a long, thin, vertical brecciated zone.

The deposit has not been worked commercially since 1941. The U. S. Army takes out minor amounts of sand from the lowest white interval for use in ash trays and flower pots.

A typical brecciated zone, located in the west end of the pit at Tip Top, Ky., was examined in some detail. The breccia fragments, composed of well-compacted white sand, range from 0.25 inch to 3 inches in diameter. The matrix surrounding the breccia fragments is limonitic and somewhat argillaceous sand, much softer than the white sand fragments. The brecciated zone is near the top of the section and lies directly below the overburden. Similar zones occur irregularly near the top of the section on all sides of the pit.

A study of the relationship of the breccia zones to the previously mentioned shale interval leads to the conclusion that the zones are fault or fracture zones which developed after deposition of the Ohio River formation. The sand was deposited, became somewhat compacted, and then later slumped into solution cavities which had developed in the underlying St. Louis limestone.

#### LABORATORY PROCEDURE

Because the Ohio River formation consists of poorly consolidated sand, channel samples rather than chip samples were taken. After an exposure had been examined, a fresh face extending from top to bottom was uncovered and a continuous channel sample was



taken. Most of the units were sampled individually for petrographic studies, but the entire exposure was sampled as one unit for beneficiation tests, as a deposit probably would be worked as a single unit if it proved to be of commercial value. Field samples averaged approximately 40 pounds in weight. Coning and quartering were used to reduce larger samples to the desired size.

#### PREPARATION

As the sand is poorly consolidated, extensive crushing is not needed. The aggregates present can be crushed by hand or by placing them in a mortar and applying very light pressure with a pestle.

An attempt was made to prevent iron contamination during preparation; a Jones splitter with steel chutes was used, however, for mixing and reducing samples. The greater accuracy that resulted from using this splitter justified the slight iron contamination which may have been introduced.

Samples were mixed and split five times to insure uniformity in texture and composition of any designated portion.<sup>1</sup> The splits were mixed by pouring one-half of them along longitudinal axis of a pan and the other half transverse to the first. Glass and porcelain pans were used exclusively in preparing samples for analysis. All samples were prepared according to the steps shown in figure 2.

#### MECHANICAL ANALYSIS

Sieve analyses were made of all samples. U. S. Standard sieves were used to make the analyses. Because the sand is well sorted, 500-gram samples were used; material larger than 0.84 millimeter was not included in the analyses. If the original sample contained material larger than 0.84 millimeter, the ratio of this portion to the total samples was calculated. The portion retained on each sieve was weighed and placed in an envelope to be used later in petrographic studies.

Samples were placed in sieves on a shaking device for different test periods. A period of 15 minutes on the shaking device seemed sufficient to separate the sample into various sieve sizes. If more than 200 grams of sand remained on any one screen after the designated time on the shaking device, the sand was run until less

<sup>1</sup> Determination of a satisfactory number of splits was made by counting small carbon rods which had been placed in a sand sample. Spectrographic determinations on limestone also have shown that five splits produce a thoroughly mixed sample.

## HIGH-SILICA SAND POTENTIALITIES

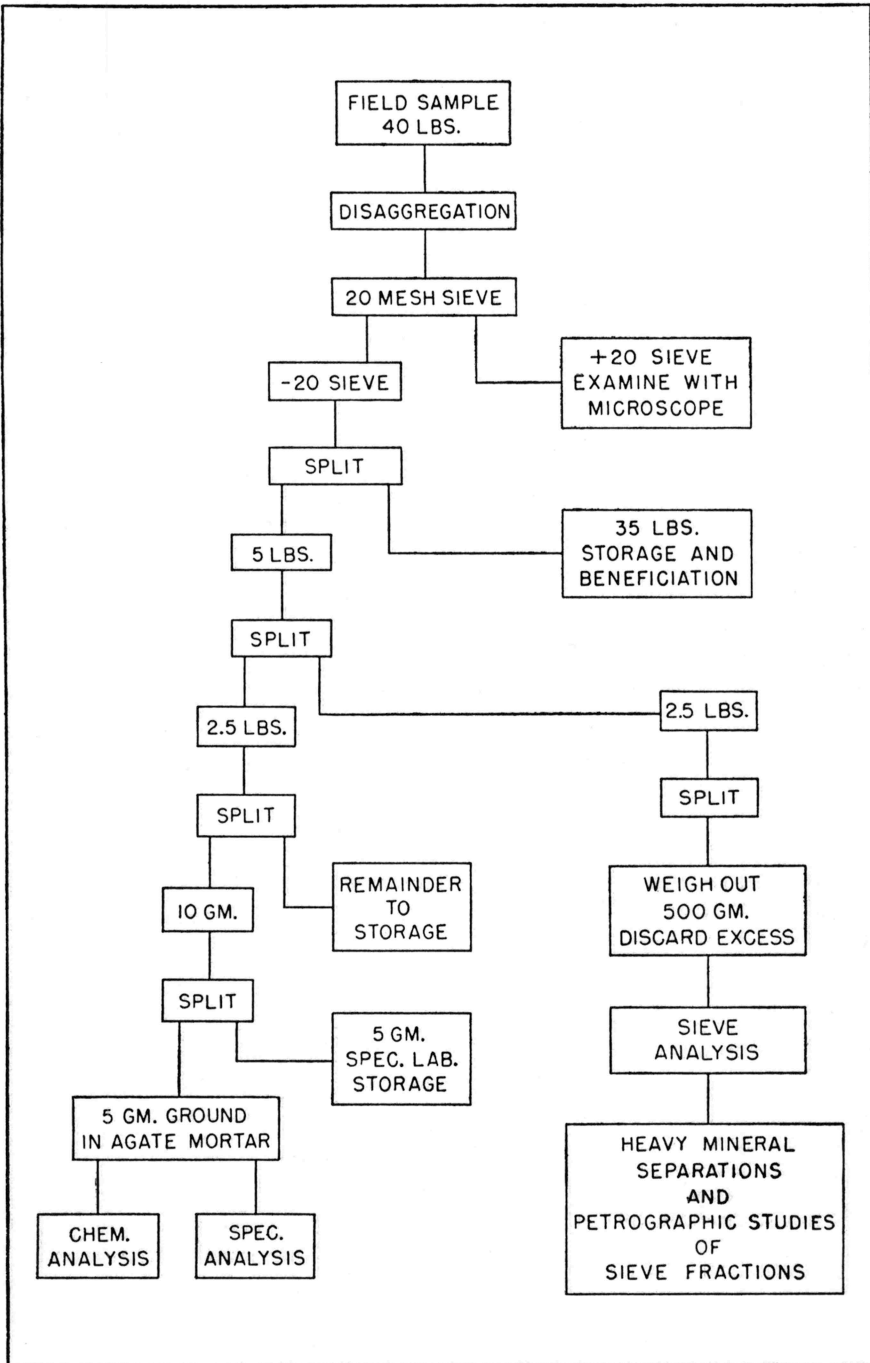


Figure 2.—Flow sheet showing procedure used in preparing samples of sand for analysis.

than 1 percent of the residue passed through the screen in 1 minute (ASTM, 1949, p. 763-765).

Two shaking devices were used: an End Shak Sieve Test Machine and a Ro-Tap Testing Sieve Shaker. As it was possible that results from the two machines might differ, their efficiencies were compared by running similar samples on each of them. The initial separation into grain sizes is faster on the Ro-Tap Testing Sieve Shaker, but after 5 minutes results from the two machines compare very closely.

#### HEAVY-MINERAL SEPARATION

Gravity separations of heavy minerals were made from each sieve size of each sample. Tetrabromoethane (sp. gr. 2.95/20°C.) was the heavy liquid used for the separations.

Samples and heavy liquid were poured into a funnel fitted at the bottom with a short length of rubber tubing and a pinch cock. The material was agitated periodically until the heavy minerals had been separated. The heavy minerals were released into a funnel fitted with filter paper and were washed with carbon tetrachloride. The grains with specific gravity less than 2.95 then were washed into a filter paper and also were washed with carbon tetrachloride. Both of the recovered portions were oven dried, weighed, and placed in bottles for further study.

#### PETROGRAPHIC ANALYSIS

Results of microscopic examination include mineral identification, percentage of minerals, shape of grains, and grain coatings. Petrographic studies were made with a Bausch and Lomb petrographic microscope. A magnification of X 75 was used for most of the work, but a magnification of X 337.5 was used when extremely high power was necessary.

Preliminary examination of the quartz residue was made with a Spencer binocular microscope at a magnification of X 42.5. Minerals were identified on the basis of their physical and optical properties. Index of refraction liquids were used as an aid in identifying minerals. Liquid with an index of refraction of 1.57 was used in mounting the minerals with a specific gravity greater than 2.95, and liquid with an index of refraction of 1.53 was used on the light minerals in order to provide ready identification of any feldspar which might be present.

The mineral percentages given here are relative percentages calculated from mounts consisting of 300 to 500 grains. Frequency numbers which in effect are logarithms of the actual mineral percentage (Evans, Hayman, and Majeed, 1933) were used. The frequency numbers have been established on the basis that a change from 5 percent to 10 percent is more significant than a change from 75 percent to 80 percent.

Grain shapes are described by employing a chart (Krumbein and Sloss, 1951, p. 81) in which sphericity and roundness of grains are shown visually. The figures range from 0.1 to 1.0, the latter figure representing a perfectly rounded and spherical grain. The sphericity and roundness of grains are expressed as an average of the portion of sample retained on each sieve. This method of expressing sphericity and roundness may seem somewhat inaccurate, but when averages of all size classes in a sample are computed, a fairly reliable figure is obtained.

Coatings on quartz grains consist of iron oxide, iron hydroxide, and various clay compounds. Differentiation between the various types of coating was not attempted. The amount of coating on quartz grains is expressed as the percentage of grain surface covered. The amount of coating was not calculated in detail, but rather the percentages given in this paper were estimated on a semiquantitative basis.

#### X-RAY POWDER DIFFRACTION ANALYSIS

Identification of the clay minerals was made with a General Electric XRD-3 X-ray Spectrometer. Nickel-filtered copper radiation was used to obtain the basal reflections.

#### RESULTS OF MECHANICAL ANALYSIS

Sand from the Ohio River formation is extremely uniform in texture and is well sorted. Results of mechanical analysis therefore show little difference from sample to sample. Variations or trends in depositional environment are not apparent from mechanical analysis. Significant variations normally would not be expected, however, since the distance between the northernmost and southernmost sampling localities is only 35 miles. Generalizations concerning the various units in the Ohio River formation cannot be drawn because even these units show no statistical variations.

Histograms showing the grain-size distribution of 10 samples of sand are shown in figure 3. These histograms appear to differ from each other, but cumulative curves demonstrate that grain-size distribution in the various samples is actually very similar.

Cumulative frequency curves were plotted on semilogarithmic paper. These curves give a better representation of grain-size distribution than histograms, as histograms of the same sample vary according to the grade scale used. Cumulative frequency curves, however, are plotted independently of grade scale, since infinitesimal grain-size variations are smoothed into a continuous curve. Cumulative frequency curves for each sample of sand are shown in figure 4. Median grain size of the sediments as well as quartile deviations also are shown.

#### SORTING COEFFICIENT

Trask (1932, p. 70-72) introduced the term sorting coefficient as a geometric measure of the ratio between the quartiles as determined on a cumulative frequency curve. Specifically it is the square root of the ratio of the quartiles; the ratio is calculated so that the value of the sorting coefficient is always greater than unity. This method of expressing geometrically the coefficient of sorting is desirable because differences in coarseness and in method of measuring various samples are eliminated. Trask stated that sorting coefficients of 3.0, 2.5, and 1.0 indicate normally sorted, well sorted, and perfectly sorted sediments respectively. Sorting coefficients of sand from the Ohio River formation range from 1.16 to 1.26, a range which indicates that the sand is exceptionally well sorted.

Geometric quartile deviation does not lend itself to direct comparison of the degree of sorting. If the sorting coefficients are expressed logarithmically, an arithmetic series results.

#### SKEWNESS

If the median grain size of a sediment coincides exactly with a point halfway between the first and third quartiles, the grain-size distribution of a sediment is perfectly symmetrical. If the distribution is not symmetrical, the curve representing grain-size distribution is said to be skewed. If the modal group (size class with the greatest concentration of sand) is coarser than the median grain size, the skewness value is less than unity. If the modal

## HIGH-SILICA SAND POTENTIALITIES

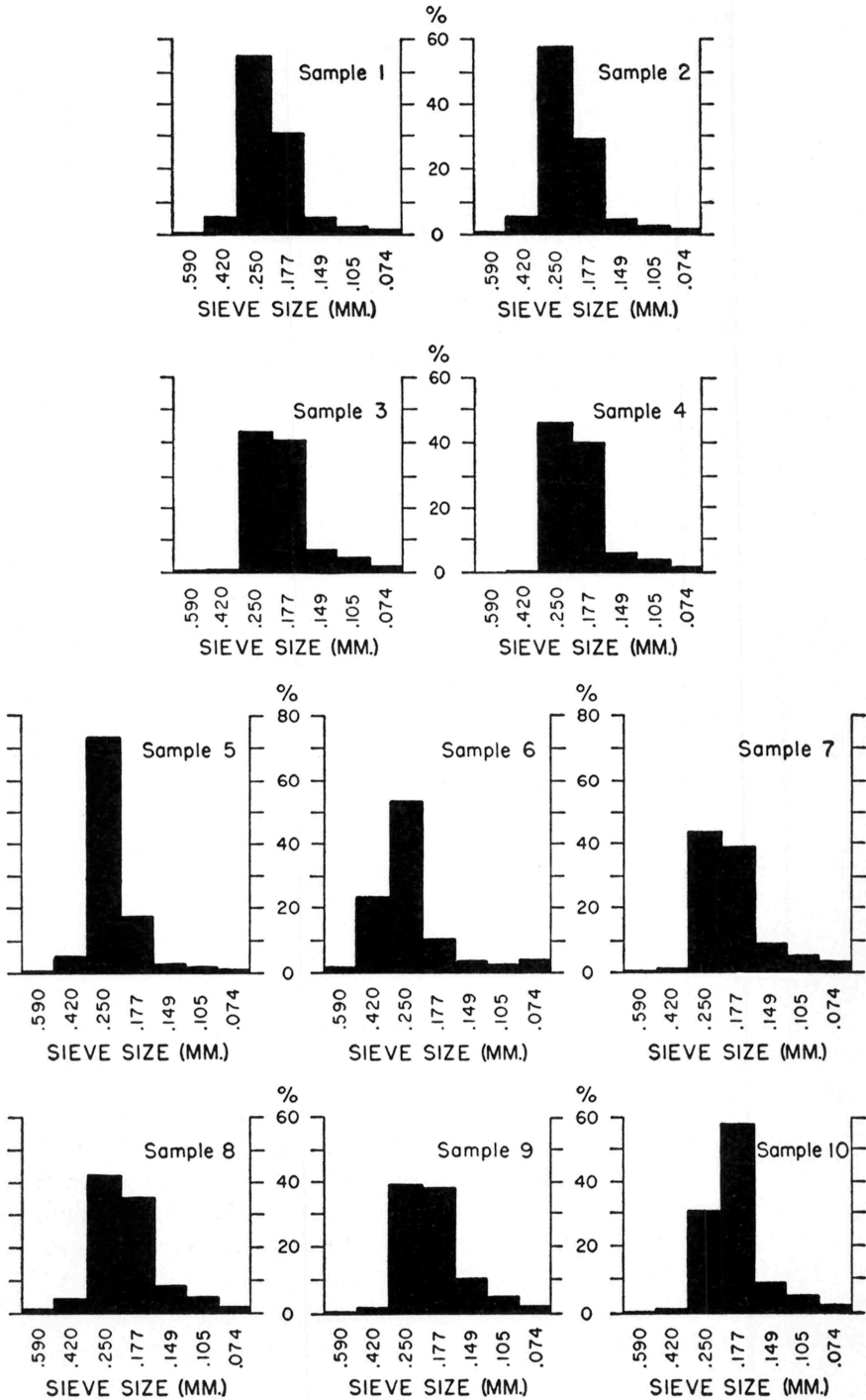


Figure 3.—Histograms showing grain-size distribution of 10 samples of sand.

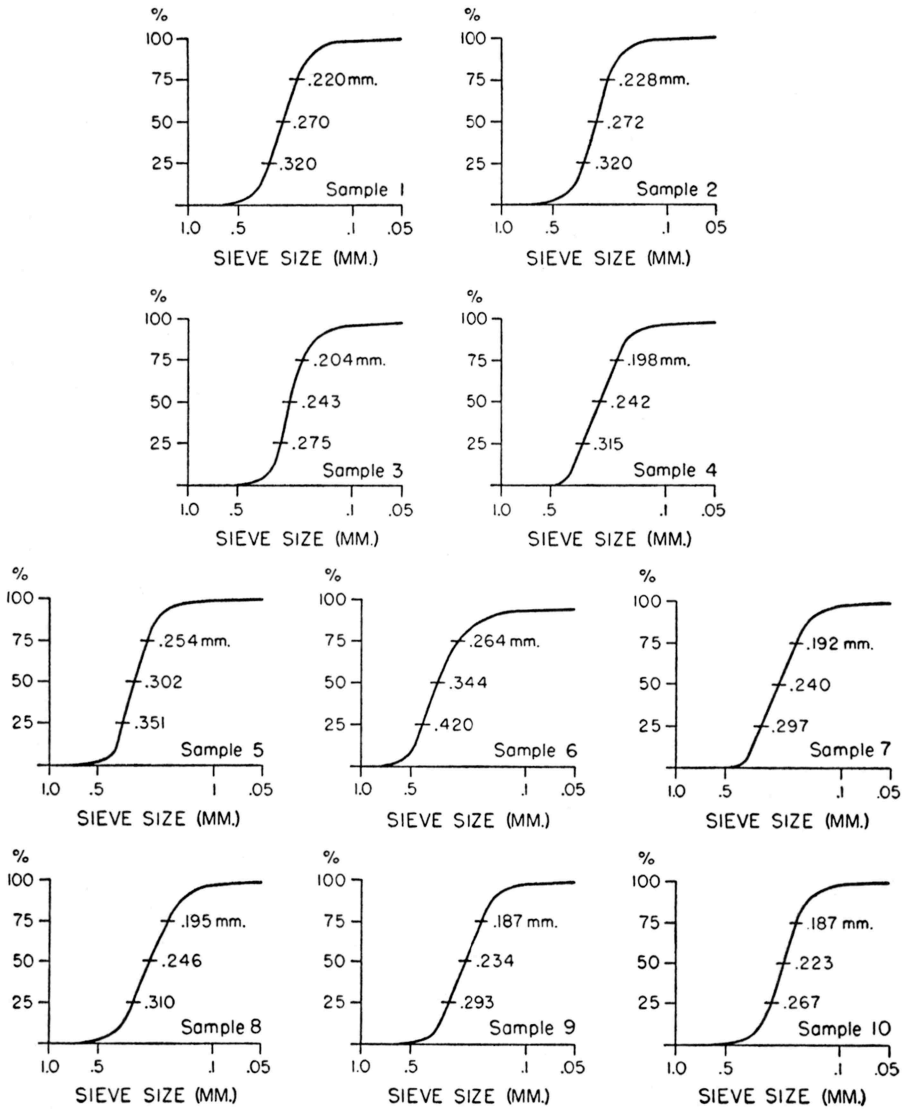


Figure 4.—Cumulative frequency curves showing median grain size and quartiles for 10 samples of sand.

group is finer than the median, the skewness value is greater than unity. The geometric measure of skewness is expressed as a square root of the ratio of the product of the quartiles to the square of the median.

Sand from the Ohio River formation is very slightly skewed toward the coarse grain size, but the skewness is so slight that the

grain-size distribution can be described as symmetrical. Very little material has been published pertaining to the use and interpretation of the skewness of sediments. From the skewness values, the generality might be made, however, that environmental conditions remained stable throughout deposition of the Ohio River formation.

Table 3 gives sorting coefficients and skewness of each sample of sand from the Ohio River formation. Logarithms for each sample also are included in order that visual comparisons can be readily made. A comparison of the logarithms demonstrates that the Ohio River formation has a very uniform texture throughout the area which was sampled for this report.

Table 3.—*Sorting coefficients and skewness of 10 samples of sand*

Sample No.	Sorting coefficient	$\log_{10} S^1$	Skewness	$\log_{10} Sk^2$
1.....	1.20	0.081	0.982	$\bar{1}.99$
2.....	1.18	0.072	0.993	$\bar{1}.99$
3.....	1.16	0.064	0.974	$\bar{1}.99$
4.....	1.26	0.100	1.020	0.01
5.....	1.17	0.068	0.988	0.00
6.....	1.26	0.100	0.968	$\bar{1}.99$
7.....	1.24	0.093	0.994	0.00
8.....	1.26	0.100	1.000	0.00
9.....	1.25	0.097	1.000	0.00
10.....	1.19	0.076	1.000	0.00

<sup>1</sup> Sorting coefficient.

<sup>2</sup> Skewness.

## RESULTS OF PETROGRAPHIC ANALYSIS

### HEAVY MINERALS

The heavy mineral suite, consisting of those minerals that have specific gravity greater than 2.95, is composed of only very stable minerals. The heavy minerals are typical of those which generally are considered to have had a granitic source (Pettijohn, 1949, p. 98). The sediments appear to have been well reworked before final deposition because the sand consists principally of quartz and stable heavy minerals. Reworking of sand from the Ohio River formation also is indicated because at least four varieties of some of the minerals have been found in the sand; this fact suggests a minimum of four distinct depositional cycles. The deposit appears to have been subjected to extensive postdepositional ground-water



activity, a fact that is evidenced by the amount of alteration of the titanium minerals and by the lack of feldspar.

Leucoxene, the most abundant mineral in the heavy-mineral residue, occurs as an alteration product of ilmenite and probably of titaniferous magnetite; its exact composition is not known. Leucoxene occurs in two varieties: (1) as hard, opaque, semilustrous, amorphous, rounded grains that range from cream to light brown in color, with hematitic and limonitic stains on some grains. Surfaces are pitted and contain grooves which commonly traverse one side of a grain. The limonitic stains occur in the pits and grooves. This variety of leucoxene is apparently a detrital form which has been transported to the site of deposition. (2) a soft earthy form, which is cream to light brown in color. The grains in this form of the mineral tend to be more angular than those in the detrital form. The earthy form has been developed in place as an alteration of the titanium minerals; it is so soft that it would be crushed and disseminated in transportation. Some of the earthy grains have a core of, or occur as a growth on, ilmenite, a mineral which is present in small amounts throughout the samples. Much of the earthy leucoxene contains small euhedral crystals of yellow lustrous rutile which protrude from the surface of the grains. This rutile is a secondary alteration product of leucoxene.

Rutile occurs as small, yellow, lustrous euhedral crystals associated with the earthy leucoxene and also as irregular, transparent orange aggregates of small grains. This latter form of rutile appears to be an intermediate stage in the development of rutile from leucoxene.

An unusual form of rutile is common in the samples from Tip Top, Ky., but is rare in the samples north of the Ohio River. This unusual form occurs as subhedral to euhedral, dark brownish-red, opaque crystals which exhibit longitudinal striae, have iridescent surfaces, and show internal reflections. It is difficult to imagine how such well-developed crystals could have been transported to the place of deposition; they undoubtedly have been formed in place.

A detrital form of rutile is present as rounded, well-polished red grains. In general, this is the most common form of rutile in the samples. A small percentage of these grains of rutile is partially covered with a thick coat of leucoxene.

Brookite occurs in minor amounts as fresh yellow crystals. Observed under a petrographic microscope, the crystals are biaxial positive with a small axial angle, are highly birefringent, and exhibit a high degree of dispersion. The brookite is not noticeably pleochroic. The mineral probably occurs as an alteration of leucoxene.

Ilmenite is present as angular to subangular fragments in all the samples. This mineral is difficult to distinguish from magnetite. Identification of ilmenite in the samples was based upon its low magnetism and upon the fact that most grains had at least a partial coating of leucoxene.

Zircon persists in variable degree throughout the samples from the Ohio River formation. It occurs as euhedral crystals, anhedral crystals, and rounded grains and ranges from colorless to pink. The most common variety of zircon is elongate, colorless, smooth, well-polished grains, which seem to indicate a long distance of transportation or possibly repeated cycles of deposition. Relatively fresh colorless grains of zircon also are present in the samples. Crystal faces are well developed, but the ends of most crystals have been fractured or broken off. Rounded, irregular, pitted, colorless grains of zircon that have rough and unpolished surfaces are rather common. Pink zircon is present but is never seen in any sample greater than 0.149 millimeter in size. The grains of pink zircon are flat and irregular in outline and have broad conchoidal fractures.

Tourmaline is one of the abundant, commonly occurring minerals and can be observed in a variety of colors: brown, green, cream to colorless, blue, pink, gray, yellow, and greenish brown. Grains range in shape from sharply angular to spherical. Inclusions are not common, but gaseous inclusions and tiny inclusions of magnetite are present.

The green, brown, and greenish-brown varieties of tourmaline are most common and are elongate and rounded to highly spherical in shape. Most angular grains occur in the light-colored varieties of tourmaline, although subhedral crystals can be observed in all the colors. All the dark-blue grains are well rounded. Fresh fractures can be seen on some spherical grains, but whether these fractures occurred during transportation of the sediment or during the mechanical analysis cannot be determined. The intensity of pleochroism in the grains varies, but, as would be expected, the

light-colored tourmaline is more strongly pleochroic than the dark variety. Authigenic tourmaline was not observed.

Muscovite, the most common mineral of the mica group, is present as very thin, unaltered colorless plates which are irregular and angular to subangular in outline. Undoubtedly, mechanical analysis broke some of the large plates into smaller fragments and caused them to appear more angular than they originally were. Tiny black inclusions occur but are uncommon in the larger muscovite plates and are virtually absent in the small flakes and fragments.

Biotite, although present in most samples, is seen only intermittently and in very minor amounts as dark-green to brown, somewhat pleochroic flakes that have subangular outlines. Alteration of biotite was not detected.

Chlorite, a very minor constituent of the heavy-mineral residue, was observed only as a few fragments in all the samples studied. It occurs as unaltered, irregular green flakes.

Limonite, the most abundant mineral in the heavy-mineral residue, occurs principally as a coating on quartz grains but also as stains on leucoxene and as irregular fragments that are probably limonitic clay. The limonitic fragments are more abundant in the coarse than in the fine sieve sizes.

Magnetite is common but occurs in minor quantities, mostly as angular crystals with traces of hematitic coating and as polished spherical grains. Magnetite is probably the most abundant mineral that occurs as small inclusions in the other minerals.

Hematite is present in minor amounts, most commonly as an oxidation product on grains of magnetite. A few red grains that resembled hematite but that had glassy luster and fracture were observed; they were considered to be siliceous hematite. Hematite also serves as cementing material which binds aggregates of fine-grained quartz. The hematite and limonite stains and coating are mostly postdepositional in origin.

Garnet is very rare in the Ohio River formation. The few grains that were seen are pink, yellow, and pale green; the pink variety predominates. The grains are clear, smooth, and very angular and have irregular conchoidal fractures. An explanation for the relative scarcity of this mineral has not been attempted in this report.

Pyrite is almost completely absent in the Ohio River formation, but one grain of pyrite was found. This grain is an aggregate of minute, euhedral pyrite crystals, too fragile to have withstood transportation; it is therefore considered to be authigenic in origin.

The heavy minerals constitute a small percentage of each sample, averaging only 0.42 percent by weight of the total. The average of the samples in Indiana is 0.21 percent and of those in Kentucky 0.75 percent. This difference in heavy-mineral content is due to the abundance of the dark-red authigenic rutile at the Tip Top deposit. The percentage of heavy minerals for individual samples is listed in table 6 (p. 46).

The heavy-mineral constituents of all samples from the Ohio River formation remain fairly constant in identity and amount except for the sporadic distribution of traces of brookite, chlorite, garnet, and pyrite. The most noticeable variable is the limonite. Differences in limonite content should be expected, however, as it is secondary in origin and imparts the different colors to the individual units. The noticeable increase in authigenic rutile is apparent in samples nos. 7, 8, 9, and 10.

Each heavy mineral variety increases in quantity from the coarse to fine sieve sizes. The metallic minerals, in general, are smaller than the median grain size of the size class with which they are retained.

Portions of the samples finer than 0.074 millimeter were not studied and therefore are not included in the mineral percentage estimates. If the sand were used industrially, this very fine sand would be removed by preliminary washing and would not be a factor in beneficiation. Since the original estimates of abundance of heavy minerals were made to only one significant number, the total mineral percentages are given to one significant number. Table 4 shows the amount of each mineral variety in the total heavy-mineral residue of each sample of sand.

Table 4.—Amount (in percent) of each mineral variety in heavy-mineral suite from 10 samples of sand<sup>1</sup>

Mineral	Sample No.									
	1	2	3	4	5	6	7	8	9	10
Leucoxene	20	20	30	50	40	40	40	20	20	20
Zircon	10	9	10	8	10	20	9	7	2	8
Tourmaline	10	10	10	10	10	10	10	6	3	7
Rutile	3	4	6	3	5	5	7	10	40	10
Muscovite	9	9	20	5	7	5	20	4	1	3
Magnetite	2	4	2	3	5	3	3	1	1	2
Ilmenite	2	T	3	2	2	2	3	2	1	2
Hematite	2	3	2	2	2	2	1	4	4	2
Limonite	40	40	10	7	5	10	4	50	30	50
Biotite	—	2	—	2	1	2	1	—	1	—
Brookite	—	T <sup>2</sup>	—	—	—	2	—	—	—	—
Chlorite	—	—	—	—	T	—	—	—	—	T
Garnet	—	—	—	—	1	T	—	—	T	—
Pyrite	—	—	—	—	—	—	—	—	—	T

<sup>1</sup> Given to one significant figure.<sup>2</sup> T—trace.

## LIGHT MINERALS

Chert is not abundant in the Ohio River formation; a maximum of 2 percent was noted in individual samples. The grains are tan to light brown, opaque, and wedge-shaped and have irregular outlines and conchoidal fracture. Surfaces of the chert grains appear to be very finely granular. This chert closely resembles that which has been seen by the author in the St. Louis formation.

Glauconite occurs as bright-green, amorphous masses that are present sporadically and in very minor amounts.

Quartz, by far the dominant mineral, constitutes at least 97 percent of the sand. Most quartz grains contain minute gaseous inclusions, which may be scattered haphazardly throughout a grain, but which are much more commonly arranged in distinct linear planes. Many of the irregular surfaces on grains have resulted from fracturing along the planes of these linear inclusions.

Some of the quartz grains are frosted, probably as the result of eolian transportation. The interior nature of these smooth, opaque grains was not determined. A few grains which are milky white, clear pink, or blue gray can be observed in any sample.

Subhedral to anhedral inclusions of tourmaline, zircon, apatite, and an opaque black mineral which is probably magnetite occur in the quartz. The number of mineral inclusions per unit volume decreases with the grain size of the quartz. Any orientation of the mineral inclusions is not apparent. The minerals generally can be

recognized in the coarse grains, but identity is uncertain, if not impossible, in grains smaller than 0.177 millimeter in diameter.

Secondary growths on quartz are not apparent in a microscopic examination of fragments. A study of the quartz in thin section, however, might reveal postdepositional enlargement of quartz grains.

*Surface texture of quartz grains.*—The surfaces of most quartz grains are smooth and unpolished but are composed of a number of smooth, irregularly shaped faces rather than one continuous surface. The grains do not appear to be pitted. Smooth frosted grains are fairly common and tend to be slightly more rounded than the average grains. A few smooth, polished grains and a small number of grains with faint, irregular striations also can be found.

*Shape of quartz grains.*—Sphericity indicates the degree to which a grain approaches the shape of a true sphere, and roundness indicates the degree to which corners and edges of grains are rounded. Sphericity and roundness are completely independent characteristics, and thus a grain having a high degree of sphericity may be highly angular according to the scale of roundness. The sphericity of quartz grains in the Ohio River formation varies with the screen size and ranges from 0.72 on the 0.42-millimeter screen to 0.7 on the 0.074-millimeter screen. Roundness of quartz grains ranges from 0.48 on the 0.42-millimeter screen to 0.25 on the 0.074-millimeter screen. Average sphericity of grains from all the sampling localities is 0.7 and average roundness of grains is 0.4.

As shown by the index numbers indicating grain shape, angularity of grains increases from coarse to fine sieve sizes. Grains within each size class, however, may range from round to angular. The sand from the Ohio River formation falls in the subround to subangular range.

#### COATING ON QUARTZ

Coatings on quartz grains consist of limonite, minor amounts of hematite, and ferruginous clay. Most of the ferruginous clay was introduced after deposition of the sand and constitutes the major part of the coatings. This secondary clay imparts most of the color to the different units in the formation. In general, the quartz grains that range from 0.42 to 0.177 millimeter in diameter

show the most coating. A progressive decrease in percentage of coated grains occurs below 0.177 millimeter.

Table 5 gives the average percentage of coated quartz grains in each sample and the percentage of those grains that are less than 10 percent coated and that are more than 10 percent coated. As relative abundances were measured, the percentages are expressed to one significant number.

Table 5.—Average percentage of quartz grains coated with limonite, hematite, and ferruginous clay in 10 samples of sand<sup>1</sup>

Sample No.	Coated (pct.)	Less than 10 percent coated (pct.)	More than 10 percent coated (pct.)
1.....	40	20	20
2.....	70	50	20
3.....	10	7	8
4.....	10	6	6
5.....	20	20	6
6.....	40	30	20
7.....	1	1	0
8.....	70	50	20
9.....	70	50	20
10.....	70	40	30

<sup>1</sup> Reduced to one significant number.

## RESULTS OF X-RAY POWDER DIFFRACTION ANALYSIS

The part of the sand finer than 0.044 millimeter from samples nos. 1, 2, and 5 (see fig. 1) was analyzed by the X-ray powder diffraction method using a recording spectrometer. The recordings were almost identical, varying only slightly in the intensity of peaks. Quartz and kaolinite proved to be the dominant minerals.

Spectrographic analysis of red sand units in the Ohio River formation shows higher iron oxide and alumina content than those of the white units. As the mineral kaolinite does not contain iron in its crystal lattice, the secondary coatings on the quartz grains must consist largely of ferruginous clay rather than a clay mineral that does contain iron.

It generally is believed that illite is dominant over kaolinite in a deposit of marine origin and that kaolinite is dominant over illite in a deposit of continental origin. The presence of kaolinite rather than illite in the sand thus supports the theory that the Ohio River formation is continental rather than marine in origin.

## BENEFICIATION

## COMMON METHODS OF BENEFICIATION

As most raw sand will not meet high-silica specifications, impurities must be removed by beneficiation. The method that is used depends upon the character and composition of impurities. Some methods of beneficiation commonly employed in industry are mentioned here.

Flotation is a method of mineral concentration whereby separation of gangue and desired material is effected either by causing the gangue to float and the desired material to remain submerged or by causing the gangue to remain submerged while the desired material floats. Even though the process is difficult to control, froth flotation is one of the most widely used commercial methods.

Wet concentrate tabling is a method of beneficiation in which separation depends upon differences in density of minerals. This method requires uniform particle size of gangue and quartz, and it usually necessitates grading of the raw sand. In order to obtain a sharp separation at least 1.0 unit of difference must exist between the specific gravities of the quartz and the impurities.

Magnetic separation is a method of beneficiation that is based upon differences in magnetic properties of minerals. The sand is passed between the poles of a magnet; the quartz is unaffected and the magnetic materials are retained by the magnet.

Water washing is generally an economical form of processing sand and is used very extensively. A water wash tends to remove clay and other finely divided particles. Washing is employed principally as a preliminary step prior to treating the sand by other methods of beneficiation.

Attrition is a process in which individual grains are allowed to abrade one another in some sort of revolving apparatus. Coating on grains is removed but grain size generally is reduced. A sand must necessarily be coarser than the desired grain size in order that this method can be applied.

Acid leaching is a method of processing that exposes sand grains to acid under controlled conditions. Stains which are not affected by water washing may react readily to the action of acids. Acid leaching is used extensively in treating sand.



Base leaching is a method of processing that exposes sand grains to caustic solutions. Alkalies are not extensively employed as a leach, but small quantities of them are added to most water washes. Sand with a high content of ferruginous clay, however, may be greatly improved by base leaching.

Beneficiation methods may be used separately, or they may be variously combined if it is economically feasible to do so.

#### METHODS APPLIED TO THE OHIO RIVER FORMATION

Several methods of beneficiating sand from the Ohio River formation have been used in making this study. The methods are laboratory methods and, although economic factors were considered, economic feasibility was not the primary criterion in choosing the methods of beneficiation that were used. Research in industrial methods must follow these laboratory investigations before the laboratory results can be applied commercially.

The analyses of raw sand in table 7 show that at least part of the Ohio River formation can be considered high-silica sand without any form of beneficiation. The sand easily meets specifications for seventh quality glass sand. The study was undertaken in order to decrease the iron content as much as possible by using simple methods of beneficiation.

As the heavy minerals constitute an average of only 0.42 percent of the sand, the possibility of beneficiation by flotation and wet concentrate tabling was eliminated immediately.

The percentages of iron oxide and aluminum oxide detract most from the purity of the sand. One important discovery made in this investigation is the fact that these substances are present as a ferruginous clay coating on quartz grains. Therefore, the most logical methods, and the ones which were adopted, were those which might effectively remove the coating on quartz. The sand was treated with a water wash, a base leach, and various acid leaches. The effects of magnetic separation also were tested.

#### METHODS OF TESTING EFFECTIVENESS OF TREATMENTS

A series of qualitative chemical tests was devised in order to determine which temperature, time interval, and acid or base concentration would produce the best results in treating a given sand. Water-washed sand samples were used in the acid leaches. The wash was performed to simulate a preliminary process that might be used commercially.

*Test with sulfuric acid.*—Ten-gram splits of a washed sample were added to equal volumes of sulfuric acid, which had been diluted to various concentrations. The acid was allowed to react on the sand for 20 minutes at room temperature; then equal volumes of the solutions were decanted into test tubes. The solutions were brought to a constant pH and were allowed to cool to room temperature. Five milliliters of a 0.6 M solution of potassium thiocyanate, which acts as an indicator of the iron in solution, were added to each test tube. Colorimetric determinations then were made with a Beckman spectrophotometer.

The acid concentration which appeared to remove the most iron was tested at various temperatures for a constant interval of time. The most effective temperature thus was determined. Sand samples prepared in the same manner as those used for the temperature tests then were tested for various intervals of time at the temperature which had given the best results. As a result of these tests, the most effective acid concentration, temperature, and time interval for treating sand with sulfuric acid were determined.

*Test with hydrochloric acid.*—Ten-gram splits of a washed sample were placed in test tubes, and 48 milliliters of acid of various concentrations were added to each 10-gram sample. The acid was allowed to react on the sand for 20 minutes and the resulting solution was decanted; then 36 milliliters of each solution were poured into clean test tubes. The solutions were brought to a constant pH, and the volumes again were made constant. After the solutions had cooled to room temperature, 5 milliliters of a 0.6 M solution of potassium thiocyanate were added to each. Colorimetric readings then were made with the spectrophotometer.

The acid concentration which gave the best results in removing iron was tested on sand samples at elevated temperatures and for various intervals of time.

Solutions of hydrochloric acid eventually attain a boiling concentration when heated for extended periods of time. The boiling concentration is a constant solution which contains 24.4 percent hydrochloric acid. Because of the instability of hydrochloric acid solutions, it was necessary to plot a boiling-point curve in order to determine the temperature at which a given solution would maintain a constant concentration.

Constant weights of sand in equal volumes and concentrations of hydrochloric acid were treated at a constant temperature for

various periods of time. The effects then were compared with the spectrophotometer, and thus the most satisfactory time interval for treating sand with hydrochloric acid was deduced.

*Test with sodium hydroxide.*—Ten grams of untreated sand were added to 80-milliliter portions of various concentrations of sodium hydroxide. The solutions ranged in concentration from 1:1 sodium hydroxide: water down to ordinary water. Each mixture was agitated with a counterrotating mixer for 5 minutes. The sand and solution were poured onto a 140-mesh (0.105 millimeter) screen. The sand retained on the screen was washed with 170 milliliters of distilled water. All liquid and the sediment finer than 0.105 millimeter were retained in a beaker, and the sediment was allowed to settle; the liquid then was decanted to the level of the sediment in the bottom of each beaker. Three drops of brom phenol blue were added to the sediment in each beaker to indicate pH. The small amount of solution remaining in each beaker was brought to a slightly acid pH with concentrated hydrochloric acid, and then 50 milliliters of a 5 N solution of hydrochloric acid were added to the solutions. The acid and sediments were allowed to react for half an hour at 100° C. in order to dissolve the contained iron. The liquid from each beaker was filtered into a test tube, and the solutions in these test tubes were brought to constant volumes and to a pH of 1.0 by the addition of distilled water and acid. Ten milliliters of solution were drawn from each test tube and diluted in 100 milliliters of water in order to decrease the percentage of iron to a concentration which could be read effectively with the spectrophotometer. Five milliliters of a 0.6 M solution of potassium thiocyanate were added to each of the dilute solutions to bring out color for the iron test. The degree of coloration was determined with the spectrophotometer, and the color of the solutions was compared with that of a standard solution in which sand had not been treated.

*Magnetic separation.*—Tests to determine the most effective separation of magnetic material were made with a Frantz Isodynamic Separator, which was set at an arbitrary degree of inclination and tilt. An unwashed sand sample was separated, each of the two parts was weighed and examined under a microscope, and then the sand was thoroughly remixed. This procedure was repeated at various degrees of inclination and tilt, while the current was kept constant. A comparison of the results of each separation led to the

determination of the inclination and tilt which would give the best magnetic separation.

#### RESULTS OF BENEFICIATION

*Water washing.*—Various methods were used to water wash samples, but the most satisfactory results were obtained by adding fresh water at the end of each 1-minute interval of agitation. After five 1-minute intervals of agitation, the water remained clear. This method can be compared to commercial methods in which fresh water is continuously circulated through the sand.

Water washing has been used in the laboratory studies discussed in this report mainly as a means of treating sand preliminary to using other methods of beneficiation. Iron content is decreased, however, by as much as one-third in some samples of sand as a result of water washing.

*Treatment with sulfuric acid.*—Samples in various concentrations of sulfuric acid were treated at room temperature. Light transmittances<sup>2</sup> were read with the spectrophotometer, converted to absorbances, and plotted against acid concentration. Absorbance values were used because they reflect iron concentration better than transmittance (Shapiro and Brannock, 1952, p. 3). The 18 N solution (1:1 acid:water) was chosen as the most satisfactory concentration for treating sand, as this concentration appears to be the minimum at which a maximum removal of iron can be obtained. The curve (fig. 5A) rises rapidly to the point corresponding to the 18 N solution and then begins to level very slightly. Sand also was treated with an 11 N solution (3:7 acid:water) in order to compare the effectiveness of iron removal of the 11 N solution with that of the 18 N solution.

Sand samples mixed with an 18 N acid solution were heated for 60 minutes at various temperatures. The best results were obtained when the mixture was heated to 140° C. (fig. 5B). Above 140° C. little change was noted in the amount of iron taken into solution. Samples then were run at 140° C. for various time intervals. Results of the test were plotted against absorbance (fig. 5C). The degree of absorbance shows a progressive increase for the first 40 minutes; after this time no appreciable change in absorbance can be seen. Thus, as a result of these tests, samples were

<sup>2</sup> Transmittance is the ratio of light incident on a solution to that leaving the solution. Absorbance equals 2-log transmittance. Absorbance is directly proportional to the depth of color in a solution, whereas transmittance is not.

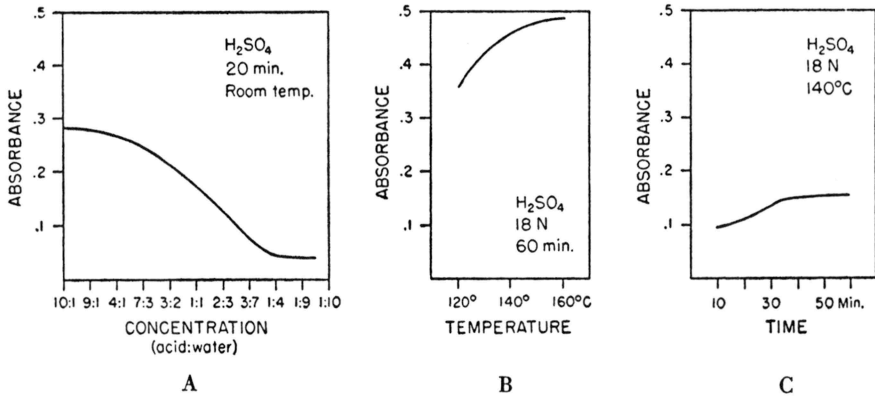


Figure 5.—Removal of iron from sand using sulfuric acid: (A) various concentrations of acid for 20 minutes at room temperature; (B) 18 N (1:1 dilution) acid for 60 minutes at various temperatures; (C) 18 N (1:1 dilution) acid for various times at  $140^\circ C$ .

treated with an 18 N solution of sulfuric acid for 40 minutes at  $140^\circ C$ .

Sand treated with sulfuric acid easily meets the specifications for second quality glass sand, and some samples of sand treated with sulfuric acid meet the requirements for first quality glass sand. Appreciable differences in the results of treating sand with 11 N, 18 N, and concentrated sulfuric acid were not evident. A preliminary magnetic separation had no effect on the final chemical analyses. The only notable difference in the chemical analyses of the samples was in the analyses of those samples which had no preliminary water wash. The alumina content of these samples was unusually high, and the sample treated with concentrated acid had an unusually high iron content.

*Treatment with hydrochloric acid.*—Tests to determine the most effective concentration of hydrochloric acid were made. The readings of percentage transmittance obtained from the spectrophotometer were converted to absorbances and plotted against acid concentration (fig. 6A). The 7 N solution (2:1 acid:water) was chosen as the concentration which seemed most suitable for treating sand with hydrochloric acid. Greater concentrations of hydrochloric acid removed a higher percentage of iron, but the 7 N solution lies at the inflection point on the curve (fig. 6A) which indicates that greater concentrations of acid do not remove a proportionally greater amount of iron. The weaker acid concentrations were used to a large extent in testing the treatment of sand

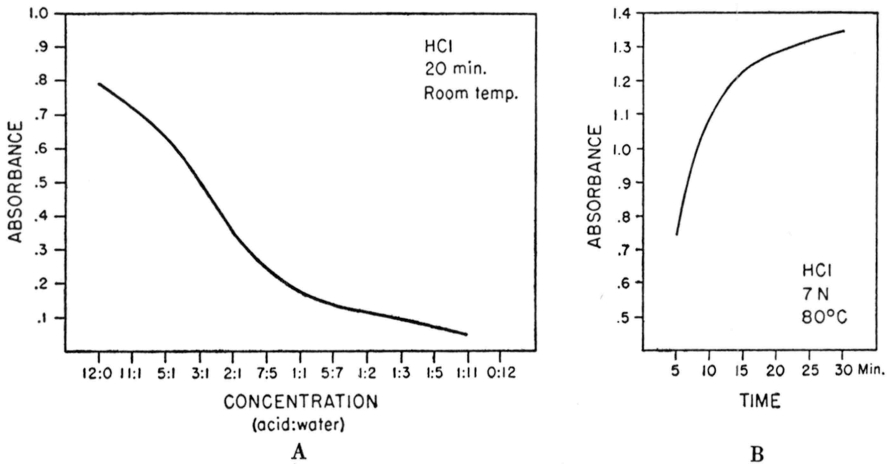


Figure 6.—Removal of iron from sand using hydrochloric acid: (A) various concentrations of acid for 20 minutes at room temperature; (B) 7 N (2:1 dilution) acid for various times at 80° C.

with hydrochloric acid because of the possible commercial application of this method of treating sand. A solution of hydrochloric acid at boiling concentration was used because hydrochloric acid is unstable at any other concentration. This boiling concentration may be more suitable commercially than other concentrations of hydrochloric acid because it is easier to control.

The maximum temperatures at which concentrations of hydrochloric acid could be run were read directly from the boiling-point curve. The 7 N solution can be raised to a temperature of approximately 90° C. without changing concentration. The boiling-point concentration can be heated to 108.5° C. before evaporation commences. Temperatures of the solutions were maintained at about 10° C. below the maximum.

The 7 N solution and the boiling concentration solution were maintained at 80° C. and 100° C. for various periods of time, and results were compared by means of the spectrophotometer and plotted (fig. 6B). The most satisfactory time interval for treating sand with hydrochloric acid at elevated temperatures is 15 minutes. The effectiveness of treatment decreases appreciably after 15 minutes. One should keep in mind that commercial suitability was a factor in choosing the concentrations of hydrochloric acid that were used in these laboratory studies.

Treatment with hydrochloric acid increased the purity of the sand appreciably. All the samples easily met the specifications

for fourth quality glass sand, and more than half of them can be considered second quality glass sand. Differences in iron content between the sand treated with 7 N and boiling-concentration solutions are only a few thousandths of 1 percent.

*Treatment with sodium hydroxide.*—The only base leach used in treating the sand was sodium hydroxide. Samples in various concentrations of sodium hydroxide were tested, and results were plotted graphically with absorbance as the ordinate and base concentration as the abscissa (fig. 7). The maximum absorbance was obtained with a 1 N solution (24:1 water:sodium hydroxide). The curve shows a progressive decrease in effectiveness of iron removal for concentrations greater than and less than the 1 N solution.

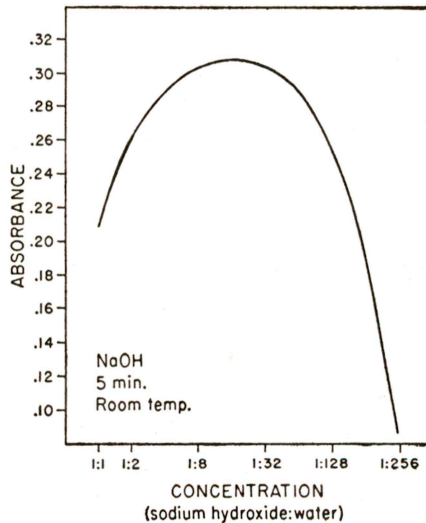


Figure 7.—Removal of iron from sand using various concentrations of sodium hydroxide for 5 minutes at room temperature.

Representative samples from all collecting localities were washed with a 1 N solution of sodium hydroxide. Although most of the samples treated with sodium hydroxide met specifications for fourth quality glass sand, the results were disappointing. Iron content is only slightly lower than that obtained by a simple water wash which had been performed in the same manner. The failure of this method of treating sand probably can be traced to the washing procedure. The sand remained in the same solution throughout

the wash; although coatings may have been loosened, they were not removed from the mixture.

One sample was washed with a 0.1 N solution of sodium hydroxide. Treatment differed from that of the 1 N solution wash in that the liquid was changed at the end of 1-minute intervals. The mineral composition of the sample washed with a 0.1 N solution compares closely with the mineral composition of the samples which were treated with the more concentrated solution. Therefore, continuous recirculation of the wash solution through the sand probably would produce a higher quality sand.

*Magnetic separation.*—Magnetic separation was not expected to decrease the iron content of the sand to any extent, but these separations were made, principally to observe their effectiveness in removing minerals which contain iron as a constituent.

Estimates based on theoretical composition were made of the amount of iron included in heavy-mineral constituents of the sand. This amount is the percentage of iron which may be removed by magnetic separation. Percentages are given for each sample in column 2 of table 6. The percentages listed under the heading "Residue" in table 6 are the differences between the total iron oxide content as determined by spectrographic analysis and the iron oxide contained in the heavy-mineral constituents of each of the sand samples. Theoretically, this residue is the amount of iron oxide that may be removed by treating the sand with an effective washing agent. Actually, however, some of the iron that is present in the heavy minerals is also removed by beneficiation.

Table 6.—Percentage of iron oxide included in composition of heavy minerals compared with total iron oxide content in 10 samples of sand

Sample No.	Heavy minerals in sample (pct.)	Fe <sub>2</sub> O <sub>3</sub> as heavy-mineral constituent in sample (pct.)	Fe <sub>2</sub> O <sub>3</sub> in spectrographic analysis (pct.)	Residue <sup>1</sup> (pct.)
1.....	0.16	0.06	0.25	0.19
2.....	0.20	0.09	0.30	0.21
3.....	0.27	0.04	0.19	0.15
4.....	0.27	0.04	0.11	0.07
5.....	0.06	0.01	0.087	0.08
6.....	0.26	0.04	0.32	0.28
7.....	0.31	0.03	0.066	0.04
8.....	0.86	0.42	0.52	0.10
9.....	1.31	0.42	0.74	0.32
10.....	0.53	0.25	0.63	0.38

<sup>1</sup> Includes iron as coatings on quartz, in interstitial clay, as inclusions in quartz, and as traces in heavy minerals which were not accounted for in the estimates.



The magnetic separation tests showed that a magnetic separator gave the best results at a 20° inclination and a 5° tilt, using a current of 1.0 ampere. Separations at inclinations of 15° and 20° gave the same results, but separations at the 15° setting took approximately twice as much time as separations at the 20° inclination. The amount of magnetic material retained dropped appreciably at inclinations greater than 20° and tilts greater than 5°. Magnetic attraction is not great enough to counteract this increase in gravity.

Magnetic separation of an unwashed sample decreased iron content 0.03 percent. In general, magnetic separations do not appreciably alter the final results when these separations are used in conjunction with other methods of treating sand. Much of the ferruginous material which can be removed by separation also can be removed by an acid leach.

*Treatment with phosphoric acid.*—When iron is introduced into a solution of phosphoric acid, a complex ion is formed. Because the iron in this complex state cannot be detected by any simple method, the effectiveness of treating sand with phosphoric acid was not tested. One sample, however, was treated with a 17 N solution (1:1 acid:water) of phosphoric acid. Results of this treatment were encouraging. Iron content was decreased sufficiently to meet the iron specifications for first quality glass sand, but alumina content was still slightly higher than specifications permit.

The composition of each sample that has been subjected to the various methods of beneficiation is given in table 7. Sample no. 1 was chosen for the initial tests because it was a typical sample and had an iron content higher than average.

Table 7.—Spectrographic analyses of raw and beneficiated sands

Sample No.	Treatment	SiO <sub>2</sub> (pct.)	Al <sub>2</sub> O <sub>3</sub> (pct.)	Fe <sub>2</sub> O <sub>3</sub> (pct.)	CaO (pct.)	MgO (pct.)	TiO <sub>2</sub> (pct.)	Na <sub>2</sub> O (pct.)	ZrO <sub>2</sub> (pct.)	MnO (pct.)
<b>Raw Sand</b>										
1.....		98.6	1.0	0.25	*0.05	0.034	0.069	*0.05	0.01	0.0003
2.....		98.6	1.0	0.30	*0.05	0.031	0.080	*0.05	0.02	0.0003
3.....		98.5	1.2	0.19	*0.05	0.039	0.080	*0.05	0.01	0.0003
4.....		98.6	1.2	0.11	*0.05	0.034	0.063	*0.05	0.01	0.0003
5.....		99.3	0.54	0.087	*0.05	0.031	0.033	*0.05	0.006	0.0002
6.....		98.4	1.0	0.32	*0.05	0.048	0.19	*0.05	0.02	0.001
7.....		98.8	0.96	0.006	*0.05	0.041	0.095	*0.05	0.02	0.0003
8.....		98.3	1.0	0.52	*0.05	0.041	0.16	*0.05	0.02	0.0006
9.....		97.9	1.2	0.74	*0.05	0.048	0.12	*0.05	0.03	0.004
10.....		97.2	2.0	0.63	*0.05	0.074	0.13	*0.05	0.01	0.003
<b>Beneficiated sand</b>										
<b>Water wash:</b>										
1.....	3 min. continuous agitation	99.35	0.40	0.17	*0.03	0.021	0.038	*0.1	0.018	0.00025
1.....	5 min. continuous agitation	99.55	0.28	0.12	*0.03	0.016	0.029	*0.1	0.0080	*0.00025
1.....	1-min. intervals of agitation until water is clear (5 min.)	99.66	0.19	0.099	*0.03	0.012	0.026	*0.1	0.011	*0.00025
<b>Magnetic separation:</b>										
1.....	raw sand	98.72	0.96	0.22	*0.03	0.031	0.046	*0.1	0.017	0.00031
1.....	water washed for 5 min. at 1-min. intervals	99.70	0.18	0.078	*0.03	0.0094	0.019	*0.1	0.0085	*0.00025
<b>Sulfuric acid:</b>										
1.....	water wash, 18 N solution, 140° C., 40 min.	99.82	0.12	0.021	*0.03	0.0077	0.022	*0.1	0.0053	0.00028
1.....	raw, 18 N solution, 140° C., 40 min.	99.61	0.32	0.022	*0.03	0.012	0.032	*0.1	0.0072	*0.00025
1.....	raw, 11 N solution, 140° C., 40 min.	99.43	0.46	0.039	*0.03	0.020	0.040	*0.1	0.011	*0.00025
1.....	raw, concentrated, 140° C., 40 min.	99.02	0.08	0.098	*0.03	0.026	0.044	*0.1	0.015	*0.00025
1.....	water wash, 18 N solution, 140° C., 40 min.	99.87	0.081	0.016	*0.03	0.0044	0.017	*0.1	0.0073	*0.00025
1.....	water wash, 11 N solution, 140° C., 40 min.	99.84	0.11	0.020	*0.03	0.0057	0.014	*0.1	0.0092	*0.00025
1.....	water wash, concentrated, 140° C., 40 min.	99.81	0.10	0.022	*0.03	0.0047	0.016	*0.1	0.0069	*0.00025
1.....	water wash, magnetic separation, 18 N solution, 140° C., 40 min.	99.81	0.088	0.015	*0.03	0.0077	0.022	*0.1	0.0053	0.00028

Table 7.—Spectrographic analyses of raw and beneficiated sands—Continued

Sample No.	Treatment	SiO <sub>2</sub> (pct.)	Al <sub>2</sub> O <sub>3</sub> (pct.)	Fe <sub>2</sub> O <sub>3</sub> (pct.)	CaO (pct.)	MgO (pct.)	TiO <sub>2</sub> (pct.)	Na <sub>2</sub> O (pct.)	ZrO <sub>2</sub> (pct.)	MnO (pct.)
<b>Sulfuric acid—Continued:</b>										
1.....	water wash, magnetic separation, 11 N solution, 140° C., 40 min.	99.79	0.15	0.021	*0.03	0.0077	0.015	*0.1	0.011	*0.00025
1.....	water wash, magnetic separation, concentrated, 140° C., 40 min.	99.84	0.12	0.028	*0.03	0.0053	0.014	*0.1	0.0075	*0.00025
2.....	water wash, 18 N solution, 140° C., 40 min.	99.77	0.16	0.032	*0.03	0.0072	0.020	*0.1	0.012	*0.00025
3.....	water wash, 18 N solution, 140° C., 40 min.	99.75	0.19	0.025	*0.03	0.0078	0.021	*0.1	0.010	*0.00025
4.....	water wash, 18 N solution, 140° C., 40 min.	99.79	0.16	0.021	*0.03	0.0067	0.017	*0.1	0.0052	*0.00025
5.....	water wash, 18 N solution, 140° C., 40 min.	99.81	0.14	0.024	*0.03	0.0067	0.014	*0.1	0.0059	*0.00025
6.....	water wash, 18 N solution, 140° C., 40 min.	99.84	0.10	0.023	*0.03	0.0059	0.022	*0.1	0.011	*0.00025
<b>Hydrochloric acid:</b>										
1.....	raw, 7 N solution, 80° C., 15 min.	99.34	0.54	0.068	*0.03	0.016	0.034	*0.1	0.0075	*0.00025
1.....	raw, 5 N solution, 100° C., 15 min.	99.39	0.54	0.11	*0.03	0.016	0.033	*0.1	0.0059	*0.00025
1.....	water wash, 7 N solution, 80° C., 15 min.	99.78	0.12	0.024	*0.03	0.0061	0.014	*0.1	0.0036	*0.00025
1.....	water wash, 5 N solution, 100° C., 15 min.	99.83	0.12	0.028	*0.03	0.0059	0.014	*0.1	0.0059	*0.00025
1.....	water wash, magnetic separation, 7 N solution, 80° C., 15 min.	99.76	0.18	0.031	*0.03	0.0082	0.014	*0.1	0.0059	*0.00025
1.....	water wash, magnetic separation, 5 N solution, 100° C., 15 min.	99.77	0.16	0.034	*0.03	0.0064	0.016	*0.1	0.010	*0.00025
2.....	water wash, 7 N solution, 80° C., 15 min.	99.64	0.26	0.050	*0.03	0.013	0.030	*0.1	0.011	*0.00025
3.....	water wash, 7 N solution, 80° C., 15 min.	99.55	0.34	0.037	*0.03	0.022	0.032	*0.1	0.018	*0.00025
4.....	water wash, 7 N solution, 80° C., 15 min.	99.57	0.34	0.035	*0.03	0.016	0.024	*0.1	0.012	*0.00025
5.....	water wash, 7 N solution, 80° C., 15 min.	99.74	0.20	0.026	*0.03	0.011	0.015	*0.1	0.0036	*0.00025
6.....	water wash, 7 N solution, 80° C., 15 min.	99.68	0.21	0.040	*0.03	0.020	0.030	*0.1	0.020	*0.00025
<b>Sodium hydroxide:</b>										
1.....	raw, 0.1 N solution, 1-min. intervals	99.82	0.10	0.050	*0.03	0.0044	0.017	*0.1	0.0053	*0.00025
1.....	raw, 1 N solution, 5 min. continuous agitation, 1 min. agitation with water	99.62	0.25	0.093	*0.03	0.0096	0.024	*0.1	0.0059	*0.00025
2.....	raw, 1 N solution, 5 min. continuous agitation, 1 min. agitation with water	99.65	0.20	0.094	*0.03	0.0095	0.032	*0.1	0.017	*0.00025

BENEFICIATION

Table 7.—Spectrographic analyses of raw and beneficiated sands—Continued

Sample No.	Treatment	SiO <sub>2</sub> (pct.)	Al <sub>2</sub> O <sub>3</sub> (pct.)	Fe <sub>2</sub> O <sub>3</sub> (pct.)	CaO (pct.)	MgO (pct.)	TiO <sub>2</sub> (pct.)	Na <sub>2</sub> O (pct.)	ZrO <sub>2</sub> (pct.)	MnO (pct.)
Sodium hydroxide—Continued:										
3.....	raw, 1 N solution, 5 min. continuous agitation, 1 min. agitation with water	99.61	0.28	0.047	*0.03	0.013	0.034	*0.1	0.018	*0.00025
4.....	raw, 1 N solution, 5 min. continuous agitation, 1 min. agitation with water	99.62	0.30	0.036	*0.03	0.013	0.024	*0.1	0.012	*0.00025
5.....	raw, 1 N solution, 5 min. continuous agitation, 1 min. agitation with water	99.71	0.21	0.039	*0.03	0.013	0.018	*0.1	0.0052	*0.00025
6.....	raw, 1 N solution, 5 min. continuous agitation, 1 min. agitation with water	99.73	0.16	0.054	*0.03	0.0080	0.036	*0.1	0.0092	*0.00025
Phosphoric acid:										
1.....	water wash, 1 N solution, 140° C., 40 min.	99.80	0.13	0.020	*0.03	0.0067	0.026	*0.1	0.015	*0.00025

\* Less than.

## CONCLUSIONS

## AGE AND ORIGIN OF THE OHIO RIVER FORMATION

Various ages and origins have been ascribed to the Ohio River formation by different workers, but a detailed study of the sand has not previously been made. The writer hoped that during his study of this formation some evidence would be found to aid in determining its age and origin.

The following observations may be useful in determining the origin of the sand:

1. The deposit is now distributed areally in a sinuous or ribbonlike pattern.

2. The haphazardly arranged crossbedding and uniform texture of the sand suggest fluvial origin.

3. The elevation of the sand above sea level progressively decreases from north to south.

4. At the east edge of the deposit in Harrison County the sand grades through silt to clay, and St. Louis limestone is found at higher altitudes than the sand. Similar phenomena were not found to the west; however, the sand and limestone may have been removed by erosion or may be under cover.

5. Fossils or fossil fragments have not been found in the formation. If it were a beach deposit, traces of fossils would ordinarily be expected. Sample no. 6 contained a few specimens of *Endothyra* and fragments of solitary and colonial corals. The fossils, however, were interpreted as being derived from the St. Louis formation.

6. The sand is extremely well sorted, and the degree of sorting does not vary from unit to unit. These are indications that conditions remained constant throughout the depositional period.

7. Symmetrical skewness curves indicate that environment remained uniform throughout deposition of the sand.

8. The heavy-mineral content is uniform laterally and vertically.

9. The varicolored units, whose colors depend upon the degree of oxidation of the contained iron, could well mark various levels

of the water table, because color is the only noticeable variable in the different units.

10. Extensive ground-water activity is suggested by the abundance of altered minerals and the lack of feldspar. Although feldspar may have been lacking in the original sediment, it also could have been present and subsequently altered to form some of the clay minerals now present.

11. The sand is found in sinkholes.

12. Breccia zones, clay-filled fractures, and displaced bedding indicate that the sand was compacted and somewhat consolidated before these features were formed, and that solution cavities into which the sand slumped had developed in the underlying limestone.

The following statement made by C. A. Malott (1922, p. 135) for an area which includes that in which the Ohio River formation lies is in accordance with the generally accepted theories: "The highest land of the Norman upland and the eastern edge of the Mitchell plain has been . . . correlated with the Highland Rim penepplain."

In summary, one may say that the Ohio River formation is a fluvial deposit which was laid down after the Highland Rim (Lexington) penepplain was in an advanced stage of development and streams were almost at grade. If the Highland Rim penepplain was developed during Pliocene time, the Ohio River formation is Pliocene in age. After deposition of the sand and during uplift of the penepplained surface, the sand was consolidated somewhat by compaction, introduction of secondary clay, and breakdown of feldspar to clay. Sinkholes were formed in the underlying limestone after uplift of the surface, and the sand slumped. Rejuvenated streams have removed a part of the deposit during the last extensive dissection of the area.

#### SUMMARY OF BENEFICIATION TREATMENTS

Impurities in the sand from the Ohio River formation are principally ferruginous clay coatings and interstitial material. The discovery of this fact about impurities, along with the fact that heavy minerals average only 0.425 percent of the sand, suggested that some acid or base leach would be the most effective method of treating the sand. Magnetic separation decreases the iron content of the raw sand by as much as 0.03 percent; acid and base leaches

seem to be as effective, however, when magnetic separation is not used as a preliminary treatment.

A preliminary water wash is essential in treating sand. Unwashed samples were not greatly beneficiated by any leaching agents that were tried.

Laboratory tests indicate that the sulfuric acid leach is the most effective and satisfactory. The instability of hydrochloric acid makes it difficult to handle and to control; therefore, it is an expensive method of beneficiation. Moreover, the purity of the sand was not increased as much with hydrochloric acid as it was with the stable, relatively inexpensive sulfuric acid.

Although results of the base leach were disappointing, base leach as a means of processing sand should not be discarded, as it would be very economical. The effectiveness of treating sand with concentrated solutions of sodium hydroxide did not reach expectations because the same solution was circulated through the sand during treatment. This fact was demonstrated by using a 0.1 N solution of sodium hydroxide, which was renewed after 1-minute periods of circulating through the sand, and which gave almost the same results as the 1 N solution. The one sample treated with phosphoric acid showed a marked decrease in iron content. Even though using phosphoric acid would be more expensive than using sulfuric acid, further investigations regarding the use of a phosphoric acid leach are advisable.

The temperatures, time intervals, and concentrations of leach solutions that were chosen were those which showed maximum iron removal with a minimum expenditure of money and energy. The proper combination of these factors gives maximum efficiency in treating sand. Spectrographic analyses indicate that slightly weaker solutions and lower temperatures also give favorable results, even though they decrease the efficiency of treatment slightly.

Tests have indicated that sand from the Ohio River formation can readily be made to meet high-silica sand specifications by using simple and inexpensive methods of beneficiation.

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