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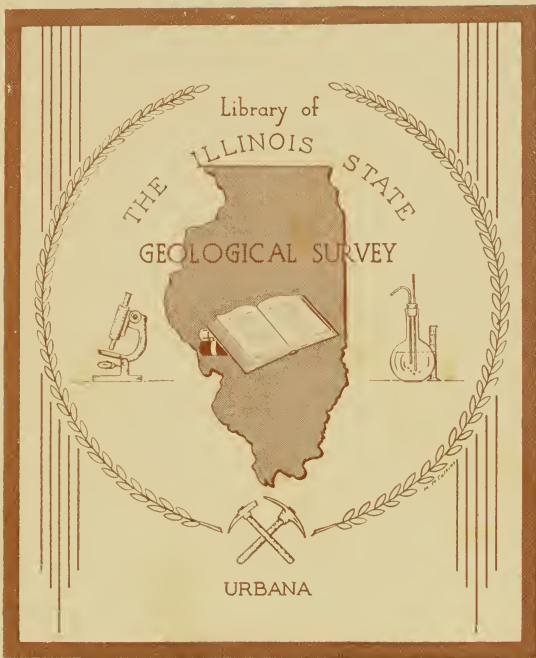
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DEPARTMENT OF REGISTRATION AND EDUCATION



# Heavy Minerals of the Cretaceous and Tertiary Sands of Extreme Southern Illinois

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# HEAVY MINERALS OF THE CRETACEOUS AND TERTIARY SANDS OF EXTREME SOUTHERN ILLINOIS

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## ABSTRACT

All of the Cretaceous and Tertiary sands of extreme southern Illinois contain a relatively uniform assemblage of heavy minerals. In general order of abundance, these heavy minerals include ilmenite and its alteration product leucoxene, zircon, kyanite, staurolite, rutile, tourmaline, sillimanite, monazite, and xenotime. The titanium minerals—ilmenite, leucoxene, and rutile—make up about 66 percent of the average heavy fraction, and  $TiO_2$  forms about 42 percent of the average heavy fraction. The heavy fractions also contain, on the average, about 7 percent  $ZrO_2$ , 0.10 percent Ce, 0.11 percent La, and 0.09 percent Y. The heavy minerals are relatively fine grained and occur mainly in the very fine sand and very coarse silt size grades.

From the standpoints of percentage of heavy minerals, volume of sand available, and sparsity of interbedded clays, silts, or gravels, the Cretaceous McNairy Formation offers far more economic interest than do the other Cretaceous and Tertiary formations. The McNairy sands are very similar in kind and proportion of heavy minerals to sands of Florida that are being exploited on a large scale primarily for their titanium minerals, but also for zircon, staurolite, and monazite. However, the Florida sands contain about 4 percent heavy minerals, whereas the McNairy sands, on the average, contain only about 1 percent heavy minerals. On the other hand, some of the McNairy sands contain considerable amounts of muscovite mica, an industrially useful mineral not found in large amounts in the Florida sands. Because of the conflicting favorable and unfavorable factors and the lack of information on the cost of mineral separation, the economic importance of the heavy minerals in the McNairy Formation is uncertain.

## INTRODUCTION

For some time it has been known that the Cretaceous sands of extreme southern Illinois contain heavy minerals, or minerals of high specific gravity, that are economically valuable if they occur in sufficiently large concentrations and in deposits of sufficiently large size (Lamar and Grim, 1937). Among the more valuable of these minerals are ilmenite, rutile, zircon, and monazite. The information on the mineralogy of the Cretaceous sands has been added to by Shrode and Lamar (1953) and Pryor (1960), and similar suites of heavy minerals have been found in the Tertiary deposits of extreme southern Illinois (Potter, 1955; Potter and Pryor, 1961; Pryor and Ross, 1962; and Frye, Willman, and Glass, 1964, p. 10-11).

The concentrations of heavy minerals in the sands generally have been considered too low to be of commercial interest. However, in view of the large amounts of sand available, the possibility of producing heavy minerals as a by-product of sand production for other uses, and the uncertainties concerning the amounts and mineralogical nature of some of the minerals, especially the opaque titanium minerals, additional work on the mineralogy of the sands was deemed appropriate.

## OCCURRENCE OF THE SANDS

The Cretaceous and Tertiary formations of extreme southern Illinois have been mapped by Weller and Krey (1939), Pryor and Ross (1962), and Ross (1964). Figure 1 shows the distribution of these deposits.

The stratigraphy, petrology, and geologic history of these formations have been discussed by many, including Lamar and Sutton (1930), Weller (1940), Lamar and Reynolds (1951), Shrode and Lamar (1953), Potter (1955), Stearns (1957), Pryor (1960), Potter and Pryor (1961), Pryor and Glass (1961), Pryor and Ross (1962), and Ross (1964). A generalized stratigraphic section of the Cretaceous and Tertiary formations of extreme southern Illinois is shown in figure 2. Of these formations, the McNairy Formation, of Cretaceous age, contains by far the most sand. The Clayton and Wilcox Formations and the "Lafayette" Gravel, all of Tertiary age, contain lesser amounts of sand. The other Cretaceous and Tertiary formations contain very little sand and are not discussed in this report.

The Cretaceous and Tertiary formations crop out in some of the steeper hillsides in the area. On more gentle slopes and on the ridge tops, they are generally overlain by loess (or windblown silt) and soil, usually less than 15 feet thick but as much as 50 feet thick near the Mississippi River (Pryor and Ross, 1962, p. 32).

## McNairy Formation

The McNairy Formation in southern Illinois consists primarily of light brown to nearly white, very fine- to fine-grained, micaceous sand. A few thin beds of sand are medium grained, with median grain diameters as coarse as 0.3 mm. Many size analyses are given by Shrode and Lamar (1953). Numerous thin beds of light gray silt to very dark gray lignitic silt and clay are interbedded with the sand. Lignitic clay and silt are predominant in the part of the formation named the Levings Member by Pryor and Ross (1962). The upper 5 to 10 feet of sand in outcrops are commonly reddish brown and clayey because of weathering.

The formation reaches a maximum thickness of 450 feet in southern Illinois (Pryor and Ross, 1962, p. 18). More than two-thirds of the total thickness of the formation in southern Illinois is sand (Pryor, 1960, p. 1498). Sand units from 10 to at least 40 feet thick with very little or no interbedded clay or silt are found throughout the outcrop belt. Near Fayville (fig. 1, and table 1, sample localities 6 and 7), for example, about 25 feet of sand is exposed, with no interbedded clay or silt; the base of the sand is not exposed. Along Post Creek (sample locality 11), about 40 feet of sand is exposed, interbedded with 5 feet of clayey silt.

Clayton Formation

The Clayton Formation in southern Illinois consists largely of clay. However, it contains sandy zones, especially at the base. The sands of the Clayton Formation contain abundant glauconite pellets.

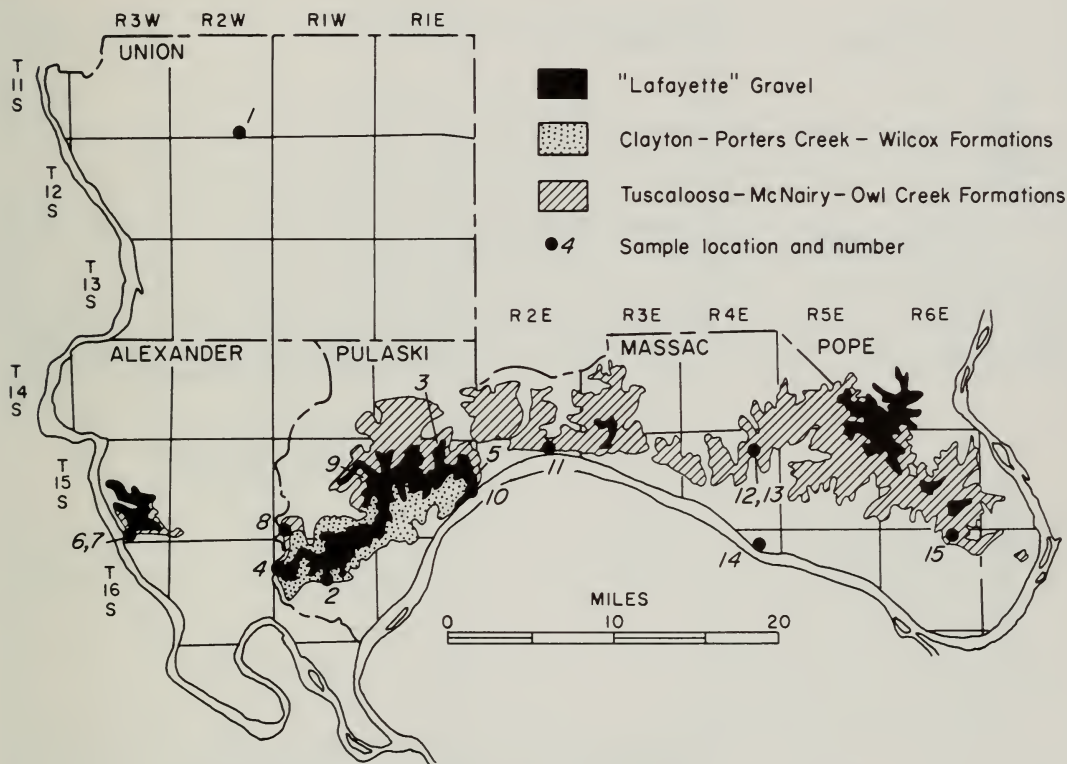


Figure 1 - Sample locations and distribution of outcropping Cretaceous and Tertiary deposits in extreme southern Illinois. Simplified from Pryor and Ross (1962), Ross (1964), and Weller and Krey (1939). The McNairy Formation comprises essentially all of the area shown as Tuscaloosa-McNairy-Owl Creek Formations. Cretaceous and Tertiary deposits covered by Pleistocene valley fill deposits are not shown. Many small areas of "Lafayette" Gravel are not shown.

Wilcox Formation

The Wilcox Formation in southern Illinois consists of interbedded clays, silts, and sands. It is poorly exposed in southern Illinois, but the limited information available suggests that sand is subordinate to clay and silt.

SYSTEM	SERIES	FORMATION	ROCK COLUMN	THICKNESS IN FEET
QUATERNARY	PLEISTOCENE	Loesses and valley fill		0 - 250
TERTIARY	PLIOCENE	"Lafayette"		5 - 50
	EOCENE	Wilcox		50 - 250
	PALEOCENE	Porters Creek		50 - 150
		Clayton		15 - 20
CRETACEOUS	GULFIAN	Owl Creek		0 - 10
		Levings Mbr.		0 - 70
		McNairy		25 - 450
		Tuscaloosa		0 - 15
Little Bear Soil				0 - 10
PALEOZOIC ROCKS				

Figure 2 - Columnar section of Cretaceous-Tertiary strata of extreme southern Illinois (from Pryor and Ross, 1962, fig. 5).

### "Lafayette" Gravel

The "Lafayette" Gravel is composed of gravel with some interbedded sand. The gravels consist chiefly of brown chert pebbles. The sands are fine to very coarse grained and are reddish to yellowish brown because of coatings of iron oxide on the sand grains. Sand is greatly subordinate to gravel in most outcrops.

### Cretaceous (?) Sand from the Kaolin District

Deposits of kaolinitic clay with small amounts of sand occur in the vicinity of Kaolin, near Anna, Illinois (sample locality 1). They have been described and assigned a questionable Cretaceous age by Lamar (1948, p. 23-29). The heavy minerals in a sample of sand from this locality (sample 1) are similar to those in sands of unquestioned Cretaceous and Tertiary age in southern Illinois.

## METHODS OF INVESTIGATION

### Sampling

Samples were collected at 15 localities where the Cretaceous and Tertiary sands are relatively unweathered and well exposed (table 1). In the thicker outcrops and in outcrops showing marked lithologic variation, samples were collected from different parts of the exposed stratigraphic section so that the variation in heavy mineral content from bed to bed could be determined. Such samples are designated by letters following the sample locality numbers in table 1. Samples were taken by cutting a continuous trench or channel from the top to the bottom of the series of beds sampled.

### Preparation of Samples for Analysis

A 50 to 100 g split of the sample was treated with hydrochloric acid to remove iron oxide cement and stains on grain surfaces. Acid-soluble iron oxide occurred in appreciable amounts only in samples from weathered portions of the McNairy Formation (samples 6, 10a, 11a, and 15a) and from the "Lafayette" Gravel (samples 2 and 3). In two samples of relatively unweathered McNairy sand in which the percentage of acid-soluble material was determined separately from the percentage of suspended fine-grained material, acid-soluble material formed less than 0.2 percent of the samples. A comparison of the heavy minerals in acid-treated and untreated portions of a sample of unweathered McNairy sand revealed no alteration caused by the acid treatment.

After acid treatment the samples were washed, and the wash water was decanted after allowing enough time, as calculated by Stokes' law, for spherical quartz grains coarser than 0.03 mm in diameter to settle from the water. Most of the finer material or "slime" remained in suspension and was lost in the decanted water together with acid-soluble material and some coarser grains, mainly mica, of disklike shape.

The amount of silt-sized heavy minerals lost in the decanted material was determined in sample 12b, which contained a larger amount (56.3 percent) of de-

TABLE 1 — LOCATION AND DESCRIPTION OF SAMPLES

Sample no.	Location							Formation	Thick-ness sam-pled (ft)	Remarks
	County	¼	¼	¼	Sec.	T.	R.			
1	Union	NE	SW	SW	35	11 S	2 W	?	3	Sand lens in clay
2	Pulaski	SE	NW	SW	15	16 S	1 W	"Lafayette"	4	Two sand lenses in gravel
3	Pulaski	NW	NE	SE	3	15 S	1 E	"Lafayette"	5	Sand lens in gravel
4	Pulaski	NE	SW	SW	7	16 S	1 W	Wilcox	3	
5a	Pulaski	SE	NW	NE	13	15 S	1 E	Clayton	1	Upper part of formation
5b	Pulaski	SE	NW	NE	13	15 S	1 E	Clayton	5	Basal part of formation
6	Alexander	NW	NW	SE	34	15 S	3 W	McNairy	4	Weathered, brownish
7a	Alexander	SE	NW	SE	34	15 S	3 W	McNairy	2	Uppermost sample
7b	Alexander	SE	NW	SE	34	15 S	3 W	McNairy	4.5	Below 7a
7c	Alexander	SE	NW	SE	34	15 S	3 W	McNairy	4.5	Below 7b
7d	Alexander	SE	NW	SE	34	15 S	3 W	McNairy	4.5	Base of exposure
8	Alexander	SE	NE	SW	31	15 S	1 W	McNairy	4.5	
9	Pulaski	SW	SW	NE	15	15 S	1 W	McNairy	5	
10a	Pulaski	SE	NE	SE	13	15 S	1 E	McNairy	3	Weathered, brownish
10b	Pulaski	SE	NE	SE	13	15 S	1 E	McNairy	7	Below 10a
11a	Pulaski	NE	NE	SW	2	15 S	2 E	McNairy	3	Weathered, brownish
11b	Pulaski	NE	NE	SW	2	15 S	2 E	McNairy	4	Below 11a
11c	Pulaski	NE	NE	SW	2	15 S	2 E	McNairy	3	Below 11b
11d	Pulaski	NE	NE	SW	2	15 S	2 E	McNairy	4	Below 11c
11e	Pulaski	NE	NE	SW	2	15 S	2 E	McNairy	3.5	Below 11d
11f	Pulaski	NE	NE	SW	2	15 S	2 E	McNairy	3	Below 2 ft of clayey silt, which is below 11e



TABLE 1 - CONTINUED

Sample no.	Location							Formation	Thick-ness sampled (ft)	Remarks
	County	¼	¼	¼	Sec.	T.	R.			
11g	Pulaski	NE	NE	SW	2	15 S	2 E	McNairy	4	Below 11f
11h	Pulaski	NE	NE	SW	2	15 S	2 E	McNairy	4	Below 11g
11i	Pulaski	NE	NE	SW	2	15 S	2 E	McNairy	5	Below 3 ft of clayey silt, which is below 11h
11j	Pulaski	NE	NE	SW	2	15 S	2 E	McNairy	2	Base of exposure
12a	Massac	NE	NE	NW	11	15 S	4 E	McNairy	11	
12b	Massac	NE	NE	NW	11	15 S	4 E	McNairy	8	Below 12a
13	Massac	NW	NW	NE	11	15 S	4 E	McNairy	6	
14	Massac	NE	SE	NE	12	16 S	4 E	McNairy	1	
15a	Massac	NW	SW	NW	2	16 S	6 E	McNairy	2	Weathered, brownish
15b	Massac	NW	SW	NW	2	16 S	6 E	McNairy	5	Below 15a

canted material than was found in any other sample. The decanted material from this sample was saved and decanted again after allowing enough time for quartz spheres coarser than 0.002 mm to settle from the water. Only 5 percent of the total amount of heavy minerals recovered from the sample was contained in the 0.002 to 0.03 mm size grade. As no other sample contained as great a percentage of decanted material as sample 12b, probably no more than 5 percent of the heavy minerals was lost by decantation in any of the samples.

After the washed sample was dried, the heavy minerals were isolated from the minerals of lower specific gravity in a separatory funnel filled with the heavy liquid tetrabromoethane (specific gravity=2.96). Despite the fine grain size of some samples, the efficiency of the separation was nearly perfect, as shown by the occurrence of only traces of heavy minerals in a fraction of intermediate density that was separated from the portion of each sample that did not sink in tetrabromoethane.

Nearly all of the muscovite mica in the samples floated in tetrabromoethane. A mica fraction was isolated from the float fraction by use of a bromoformacetone mixture adjusted to a specific gravity of 2.7. The "sink" fraction of this separation, or the intermediate fraction of the whole sample, consisted almost entirely of muscovite mica. The "float" fraction, or the light fraction of the whole sample, consisted predominantly of quartz. Some mica remained in the light fraction. However, examination of this mica with the petrographic microscope revealed that most of it is partially altered to expanded, vermicular aggregates or "books" consisting

presumably of clay minerals. Therefore, its presence in this fraction is probably due to its specific gravity being lower than that of unaltered muscovite.

Somewhat greater percentages of mica can be obtained from the samples by froth flotation than by heavy liquid separation. The froth flotation method resulted in the separation of 4.67 and 7.76 percent mica from samples 8 and 9 (Larry R. Camp, personal communication), whereas the heavy liquid method resulted in the separation of 4.1 and 6.9 percent mica from the same two samples.

### Mineralogic Analyses

The heavy fraction of the sample was split to a size suitable for examination by petrographic microscope, and the grains were mounted in an immersion oil having an index of refraction of 1.70. From 200 to 300 grains per sample were counted by the point-count method, and their diameters were measured by use of the micrometer ocular.

Mineral frequencies by number of grains were converted to weight percentages by a modification of the method described by Hunter (1967). The modifications were as follows:

1. the weight of a grain was assumed to be proportional to the product of three mutually perpendicular axes (length, width, and thickness) rather than to the cube of the grain width, and
2. the probability of encountering a grain by the point-count method was assumed to be proportional to the product of length and width rather than to the square of the width.

Given these modified assumptions, it can be shown that the density-grain shape weighting factor for a mineral ( $\rho_i$  in equation 5 of Hunter, 1967, p. 524) is equal to the mineral density multiplied by the average ratio of grain thickness to grain width of the mineral.

The average ratios of grain thickness to grain width were determined from measurements of twenty grains of each mineral. Grain thicknesses were determined by focusing the microscope first on the top of the grain and then on the flat surface on which the grain rested, and by measuring the distance between the two focal planes by use of the fine focusing knob of the microscope. The thickness measurements made in immersion oil were corrected to give values equal to those made in air.

Values of the density-grain shape weighting factors for the heavy minerals are given in table 2.

## MINERALOGIC COMPOSITION OF THE SANDS

### Decanted Fraction

The material decanted in wash water after acid treatment makes up from 2.4 to 56.3 percent of the samples (table 3). This material consists mainly of clay and silt finer than 0.03 mm in grain diameter, with small amounts of mica coarser than 0.03 mm and, especially in samples from the "Lafayette" Gravel and weathered por-

tions of the McNairy Formation, acid-soluble iron oxides. The clay minerals in the Cretaceous and Tertiary deposits of extreme southern Illinois have been described by Pryor and Glass (1961).

#### Light Fraction

Light minerals (specific gravity less than 2.7) make up from 42.9 to 97.0 percent of the samples (table 3). This material consists largely of quartz. The quartz and other light minerals of the Cretaceous and Tertiary sands have been described by Pryor (1960, p. 1493-1496) and Potter and Pryor (1961). Graphite occurs in small amounts in the light fractions of sands from the McNairy Formation (Shrode, 1954; Pryor, 1960, p. 1494). The largest graphite content reported by Pryor is 1 percent of the sand. Glauconite occurs abundantly in the Clayton

TABLE 2 - DATA USED IN CONVERTING MINERAL FREQUENCIES BY NUMBER OF GRAINS TO WEIGHT PERCENTAGES

Mineral	Density (g/cm <sup>3</sup> )	Average thickness/width/length	Density-grain shape weighting factor*
Tourmaline	3.2	.75 / 1 / 1.7	2.4
Sillimanite	3.2	.71 / 1 / 2.1	2.3
Kyanite	3.6	.51 / 1 / 1.9	1.8
Staurolite	3.7	.72 / 1 / 1.4	2.7
Rutile	4.2	.76 / 1 / 1.4	3.2
Leucoxene	4.0	.64 / 1 / 1.3	2.6
Ilmenite	4.5		2.9
Zircon	4.7	.78 / 1 / 1.7	3.7
Monazite	5.1	.75 / 1 / 1.3	3.8

\*Density multiplied by average thickness/width. This weighting factor is  $\rho_i$  in equation 5 of Hunter (1967, p.524).

Formation. Because the specific gravities of the glauconite grains are in part greater and in part less than 2.7, intermediate fractions were not separated from the Clayton samples and the glauconite is included in the light fractions (table 3, samples 5a and 5b).

#### Intermediate Fraction

Material of intermediate specific gravity (from 2.7 to 2.96) makes up from a trace to 6.9 percent of the samples (table 3). This material consists almost entirely of muscovite mica. Of the sands studied, only the McNairy Formation contains large amounts of muscovite. Pryor (1960, p. 1494) reports that beds as much as 3 inches thick composed entirely of muscovite have been observed in the McNairy Formation. Average muscovite contents of McNairy Formation sands reported

by Pryor (1960, p. 1493-1494) are 8.43 percent as determined by point counts of seven thin sections and 3.04 percent as determined by grain counts corrected for particle shape effects.

In the sample containing the most mica (sample 9), 95 percent of the mica is in the size range from 200-mesh (0.073 mm) to 48-mesh (0.295 mm).

Heavy Fraction

Most of the samples contain less than 1 percent heavy minerals, and the highest content found was 1.4 percent (table 3). The highest percentages occur in the McNairy Formation. The sands of the Clayton Formation also contain rela-

TABLE 3 - MINERALOGIC COMPOSITION OF SAMPLES

Sample no.	Composition of sample (weight percent)				Composition of heavy fraction of sample (weight percent)								
	Decanted fraction*	Light fraction**	Inter-mediate fraction†	Heavy fraction	Tourmaline	Sillimanite	Kyanite	Staurolite	Rutile	Leucocoxene	Ilmenite	Zircon	Monazite and Xenotime
1	7.6	92.3	Tr	0.1	14		3	4	3	65	9	3	
2	11.4	88.4	Tr	0.2	11	3	25	9	2	15	31	5	
3	8.5	91.4	Tr	0.1	15		0.9	0.9	2	61	3	17	
4	10.1	89.8	Tr	0.1	13	0.5	6	10	3	36	16	15	
5a	31.7	67.5	‡‡	0.8	5	0.4	9	5	4	13	47	16	2
5b	41.8	57.9	‡‡	0.3	7	0.8	10	16	3	18	38	7	0.5
6	5.8	93.1	0.6	0.5	4	0.4	7	8	4	39	26	11	0.7
7‡	6.7	92.5	0.4	0.4	5	1	12	9	4	43	11+	12	3
7a	14.2	83.6	1.4	0.8									
7b	5.9	93.4	0.1	0.6									
7c	5.7	93.8	0.3	0.2									
7d	5.0	94.5	0.3	0.2									
8	34.4	60.9	4.1	0.6	5	1	8	5	3	30	33	13	0.8
9	5.1	86.9	6.9	1.1	6	2	2	3	5	53	19	9	0.4
10a	15.3	84.0	0.1	0.6	6	3	10	15	4	17	41	3	0.5
10b	4.6	94.3	0.4	0.7	6	2	10	12	5	14	38	12	2
11‡	8.0	90.5	0.8	0.7	5	1	10	7	5	35	25	11	1
11a	8.0	90.2	0.9	0.9									
11b	33.1	65.8	0.5	0.6									
11c	4.4	94.7	0.1	0.8									
11d	2.8	96.7	0.3	0.2									
11e	2.4	97.0	0.3	0.3									
11f	2.4	96.1	1.0	0.5									
11g	5.1	93.1	0.8	1.0									
11h	2.9	95.5	0.5	1.1									
11i	6.6	91.2	1.5	0.7									
11j	10.8	86.4	2.2	0.6									
12‡	51.8	46.9	0.7	0.6	4	0.5	7	5	8	40	19	15	2
12a	48.7	49.7	0.8	0.8									
12b	56.3	42.9	0.5	0.3									
13	30.8	66.2	1.6	1.4	3	1	6	5	5	39	24	17	0.4
14	33.9	64.9	0.4	0.8	2	2	9	9	9	18	37	12	1
15a	18.9	79.8	0.4	0.9	5	1	6	6	8	34	28	11	1
15b	11.5	86.9	0.8	0.8	4	1	7	7	6	40	21	12	1

\*The decanted fraction consists largely of clay and silt finer than 0.03 mm in grain diameter, with smaller amounts of acid-soluble iron oxides.

\*\*The light fraction consists largely of quartz.

†The intermediate fraction consists largely of muscovite mica; Tr = trace.

‡Composite sample.

‡‡Glauconite, some of which is of intermediate density, is included in light fraction.

tively high percentages, but the samples from the other formations contain very small amounts of heavy minerals. Within the McNairy Formation, no consistent geographic or stratigraphic variations in percent heavy fraction were noted.

All of the Cretaceous and Tertiary formations of extreme southern Illinois have a relatively uniform assemblage of heavy minerals. The minerals are described in the following paragraphs in order of their probable economic importance.

#### Ilmenite ( $\text{FeTiO}_3$ ) and its Alteration Product, Leucoxene

Ilmenite and leucoxene are the main sources of titanium dioxide for white pigment and may also serve as a source of titanium metal for aeronautical use. Sands are important sources of these minerals.

Ilmenite and leucoxene form a gradational series in the sands studied. Apparently unaltered ilmenite grains are black, opaque, and have a metallic or sub-metallic luster. Grains completely altered to leucoxene are white or pale yellowish or brownish, translucent in intense convergent light, and generally have a dull luster. Most of the grains are intermediate in character between these two extremes; they vary from yellow or light brown to dark brown in color and commonly have opaque cores and translucent rims. The darker grains were classified as ilmenite, the lighter grains as leucoxene.

The black opaque grains are the most magnetic of all the grains in the ilmenite-leucoxene series (fig. 3). Examination of a polished grain mount of the most magnetic fraction by a petrographic microscope using vertically incident light showed that these grains have the optical properties of ilmenite; they are monocrystalline, anisotropic, and are pinkish gray in reflected light. An X-ray diffractogram of a powdered sample of the most magnetic fraction confirmed the presence of ilmenite.

The white translucent leucoxene grains are the least magnetic grains in the series (fig. 3). A polished grain mount of the least magnetic fraction showed that the leucoxene grains are aggregates of very finely crystalline particles. The particles are anisotropic, white or light gray in reflected light, and show reddish brown or yellowish internal reflections, but are too small to be definitely identified optically. Most of the aggregates are crystallographically unoriented, but some are oriented so that the constituent particles tend to extinguish simultaneously under crossed nicols.

The mineralogical nature of leucoxene has been the subject of considerable investigation. It is now generally agreed that leucoxene is a term that has been applied to a variety of very finely crystalline, titanium-rich substances; it is most commonly rutile, but forms consisting of anatase, brookite, and sphene are also reported (Allen, 1956; Bailey et al., 1956; Bailey and Cameron, 1957; Creitz and McVay, 1948; Golding, 1961; Houston and Murphy, 1962; Lynd, 1960; Temple, 1966; Tyler and Marsden, 1938). In addition, an alteration product intermediate between ilmenite and leucoxene has been named pseudorutile (Temple, 1966). An X-ray diffractogram of a powdered sample of a slightly magnetic fraction (magnetic at settings of  $5^\circ$  side tilt and 1.2 amp on the Frantz Isodynamic Separator and non-magnetic at settings of  $15^\circ$  and 0.75 amp) that consisted almost entirely of leucoxene (fig. 3) revealed the presence of both rutile and anatase; the principle peaks of the two minerals on the diffractogram were of similar height.

Polished grain mounts of intermediate magnetic fractions showed various stages in the alteration of ilmenite to leucoxene. Some of the leucoxene occurs in well defined zones in grain rims, in irregular veinlets, and along cleavage planes

of ilmenite grains. Some leucoxene, on the other hand, occurs finely disseminated through ilmenite grains. The ilmenite with finely disseminated leucoxene is light gray in reflected light, rather than pinkish gray as is the unaltered ilmenite, and is slightly less anisotropic than the unaltered ilmenite. Very little of the slightly anisotropic to isotropic alteration product described by Bailey et al. (1956) or the dark gray, weakly reflective anisotropic material described by Temple (1966) was found in these sands.

Ilmenite and leucoxene, together, comprise the most common heavy constituent of the sands. They make up from 46 to 74 percent of the heavy fraction and, in the McNairy Formation samples, average 60.3 percent. In contrast to the relative uniformity in the ratios of the other components to each other and to the sum of leucoxene plus ilmenite, the ratio of ilmenite to leucoxene varies greatly

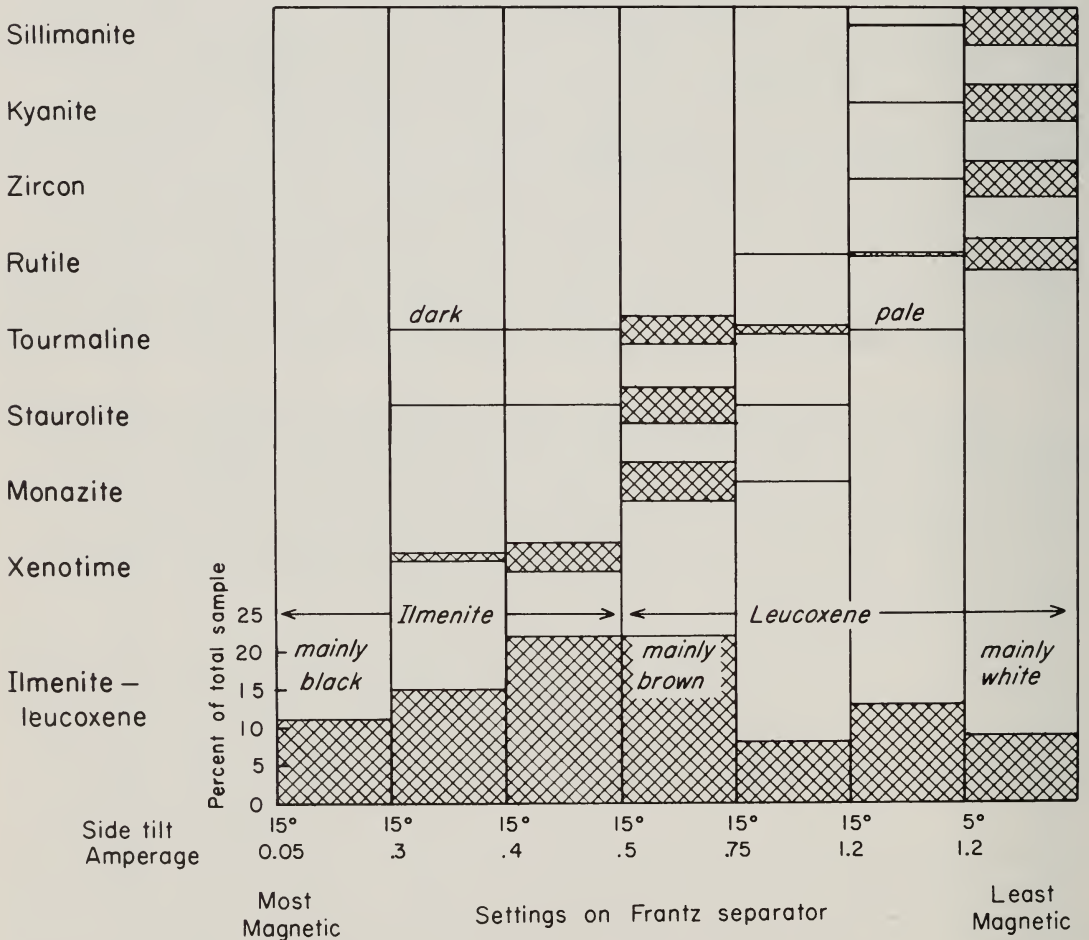


Figure 3 - Magnetic properties of the heavy minerals of the McNairy Formation of extreme southern Illinois. A composite sample of all samples collected from the formation was used in determining the magnetic properties. The forward tilt on the Frantz Isodynamic Separator was 25°.

from sample to sample (table 3). This variation suggests that at least most of the alteration of ilmenite to leucoxene took place after deposition of the sand. An increase in the ratio of ilmenite to leucoxene with increasing depth in deposits at Trail Ridge, Florida, led Temple (1966) to conclude that the alteration was caused largely by post-depositional weathering.

#### Rutile ( $\text{TiO}_2$ )

Rutile is the chief source of titanium metal and is also widely used as welding-rod coatings. It is present in small to moderate amounts in the heavy fractions, ranging from 2 to 8 percent (table 3), and in the McNairy Formation samples averages 5.5 percent of the heavy fraction.

#### Zircon ( $\text{ZrSiO}_4$ )

Zircon is used largely as a refractory; it is also the source of zirconium. It is a common mineral in the sands, comprising from 3 to 17 percent of the heavy fraction (table 3). In the McNairy samples it averages 12.6 percent.

#### Monazite [ $(\text{Ce}, \text{La}, \text{Y}, \text{Th})\text{PO}_4$ ] and Xenotime ( $\text{YPO}_4$ )

Monazite is an important source of rare earth elements. It commonly contains enough thorium to serve as a source of this radioactive element and also contains small amounts of uranium (Mertie, 1960). Xenotime is a source of yttrium and related elements. Monazite and xenotime occur in small amounts (0 to 3 percent) in the heavy fractions. In the McNairy samples, the average content is 1.1 percent. Xenotime makes up less than one-fifth of these percentages. Distinguishing between monazite, xenotime, and zircon by their indices of refraction was relatively simple in the high-index immersion oil used. Both the monazite and the xenotime are very pale yellowish or greenish in color. The two minerals can be separated magnetically (fig. 3).

#### Staurolite [ $\text{Fe}_2\text{Al}_9\text{O}_7(\text{SiO}_4)_4(\text{OH})$ ]

Staurolite is recovered as a by-product in the separation of titanium minerals from sands at Trail Ridge, Florida, and is sold as a source of alumina for cement (Carpenter et al., 1953). In the southern Illinois samples, staurolite makes up from 1 to 16 percent of the heavy fraction (table 3). The heavy fractions of the McNairy samples contain an average staurolite content of 6.5 percent. The staurolite in these sands is variable in color; the lighter colored staurolite is of the optically negative variety described by Drysdall and Stillman (1963).

#### Kyanite and Sillimanite ( $\text{Al}_2\text{SiO}_5$ )

Kyanite and sillimanite are used in refractories. Kyanite is a common heavy mineral in the sands, forming from 1 to 25 percent of the heavy fraction (table 3) and averaging 8.0 percent in the samples from the McNairy Formation. Sillimanite is much less common than its polymorph, kyanite. Its content in the heavy fractions ranges from 0 to 3 percent and, in the McNairy samples, averages 1.4 percent.

#### Tourmaline [ $\text{Na}(\text{Mg}, \text{Fe})_3\text{Al}_6(\text{Si}_6\text{O}_{18})(\text{BO}_3)_3(\text{OH})_4$ ]

The tourmaline in the southern Illinois sands has no known economic value. It makes up from 2 to 15 percent of the heavy fraction. In the heavy fractions of

the McNairy Formation samples, the average percentage is 4.5. The types of tourmaline occurring in the sands are described by Pryor (1960, p. 1495).

#### Others

Heavy minerals found in trace amounts in one or more samples include green spinel, chloritoid, anatase, epidote, and garnet. Jarosite, identified by X-ray diffraction analysis, occurs as spherulitic and very finely granular aggregates in the samples (5a and 5b) from the Clayton Formation. The jarosite probably formed by a reaction between glauconite and acidic sulfate-bearing solutions from the weathering of pyrite; this process has been suggested by Tyler (1936), Briggs (1951), and Mitchell (1962). Heavy minerals found in small amounts by other workers (Lamar and Grim, 1937; Potter, 1955, p. 10-11; and Pryor, 1960, p. 1495) include andalusite, enstatite, hypersthene, hornblende, tremolite, topaz, and magnetite.

### GRAIN-SIZE DISTRIBUTIONS OF THE MINERALS

The heavy fractions of the sands are consistently finer grained than the rest of the material. Most of the heavy fractions have median grain sizes in the very fine sand or very coarse silt grade (table 4). The more dense or "heavier" heavy minerals, zircon and monazite, are finer grained than the other heavy minerals, whereas the less dense heavy minerals, tourmaline, kyanite, and staurolite, are more common in the coarser size grades (figs. 4 and 5). The median grain size of the ilmenite-leucoxene in the McNairy Formation samples is nearly the same as that of the whole heavy fractions and is approximately 1/16 or 0.0625 mm (fig. 5). Size data on the heavy minerals of the "Lafayette" Gravel are given by Potter (1955, p. 8-10).

TABLE 4 — MEDIAN GRAIN SIZES OF HEAVY FRACTIONS AND LIGHT PLUS DECANTED FRACTIONS OF THE SAMPLES

Sample no.	Median grain diameter (mm)	
	Light plus decanted fraction*	Heavy fraction†
1	.21	.11
2	.29	.14
3	.22	.11
4	.45	.10
5a	.12	.069
5b	.13	.10
6	.10	.047
7	.13	.063
8	.072	.054
9	.077	.051
10a	.18	.095
10b	.18	.12
11	.16	.077
12	<.044	.043
13	.051	.047
14	.091	.072
15a	.10	.054
15b	.11	.058

### CHEMICAL COMPOSITION OF THE SANDS

Titanium and zirconium in the heavy fractions of selected samples were analyzed by X-ray fluorescence, and cerium, lanthanum, and yttrium were analyzed by optical emission spectrography.

The  $TiO_2$  contents of the analyzed heavy fractions range from 33.8 to 50.6 percent (table 5). These values agree relatively well with the  $TiO_2$  percentages estimated from the mineralogical analyses given in table 3. The  $TiO_2$  percentages were estimated by adding the estimated  $TiO_2$  contributed

\*For the purpose of determining median grain diameter, it was assumed that the decanted fraction is composed entirely of suspended material finer grained than 270-mesh (0.053 mm). The median grain diameter of the light plus decanted fraction is approximately the same as that of the whole sample.

†The microscopically measured grain diameters were converted to equivalent sieve size grade by the procedure outlined by Hunter (1967).



by the titanium minerals rutile, leucoxene, and ilmenite. The  $TiO_2$  contents of the ilmenite and leucoxene are poorly known, as both are made up of grains of varying compositions. On the basis of published analyses of ilmenite and leucoxene from sand deposits (Creitz and McVay, 1948; Houston and Murphy, 1962; Temple, 1966) and mineralogical analyses of polished grain mounts, the leucoxene was estimated to contain 70 percent  $TiO_2$  and the ilmenite 55 percent  $TiO_2$ .

The weight percentage of recoverable  $TiO_2$  in the whole sample was calculated from the analysis of the heavy fraction (table 5) and the percent heavy fraction in the sample (table 3). It was assumed that no recoverable  $TiO_2$  occurs in the light and intermediate fraction. The estimated recoverable  $TiO_2$  contents range from less than 0.1 to 0.6 percent (table 6). All values greater than 0.3 percent were from the McNairy Formation.

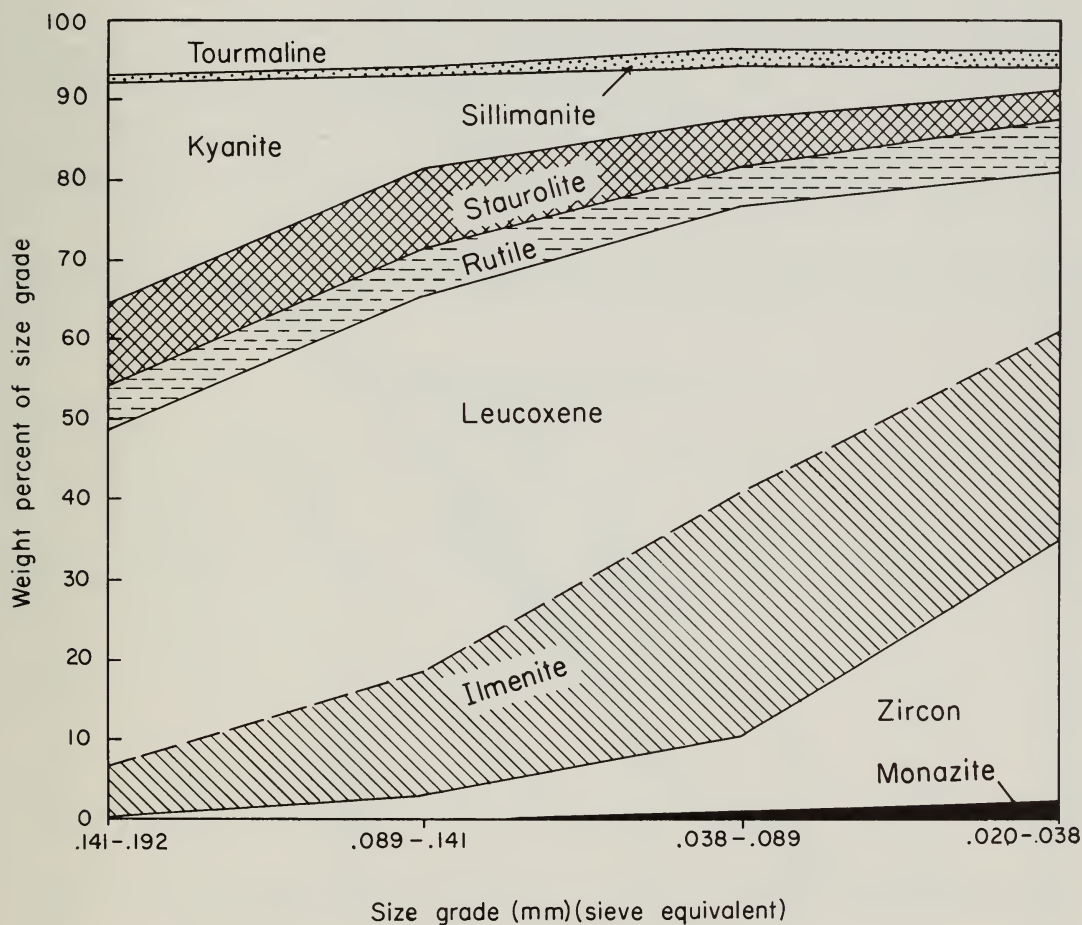


Figure 4 - Average mineralogic composition by size grade of the heavy fraction of sand from the McNairy Formation of extreme southern Illinois. The average is an unweighted average of all samples. The microscopically measured grain diameters were converted to equivalent sieve size grade by the procedure outlined by Hunter (1967).

The  $ZrO_2$  contents of the analyzed heavy fractions range from 4.5 to 10.7 percent (table 5). These values are fairly similar to the  $ZrO_2$  percentages estimated from the mineralogical analyses by assuming that the zircon was pure  $ZrSiO_4$  containing 67.2 percent  $ZrO_2$  (table 5). The estimated recoverable  $ZrO_2$  contents of the whole samples, calculated in the same way as recoverable  $TiO_2$ , range from a trace to 0.1 percent. All values higher than 0.06 percent occur in the McNairy Formation samples.

The sums of the Ce, La, and Y contents of the analyzed heavy fractions range from 0.08 to 0.64 percent (table 5). The average contents are 0.10 percent Ce, 0.11 percent La, and 0.09 percent Y.

The percentages of monazite and xenotime may be estimated from the chemical analyses. Monazite is of variable composition, but a number of analyses (Wylie, 1950; Murata, Rose, and Carron, 1953; Murata et al., 1959; Heinrich, Borup, and Levinson, 1960; Mertie, 1960; Deer, Howie, and Zussman, 1962, p. 339-346; and Flinter, Butler, and Harrel, 1963) suggest an average of approximately 13 percent  $La_2O_3$  and 27 percent  $Ce_2O_3$ , or 11 percent La and 23 percent Ce. Assuming a cerium plus lanthanum content of 34 percent for the monazite in these sands, the average calculated monazite content of the analyzed heavy fractions is 0.6 percent. Assuming that the xenotime is pure  $YPO_4$  containing 48 percent Y, the average calculated xenotime content of the analyzed heavy fractions is 0.2 percent. The sum of the two, 0.8 percent, is similar to the average con-

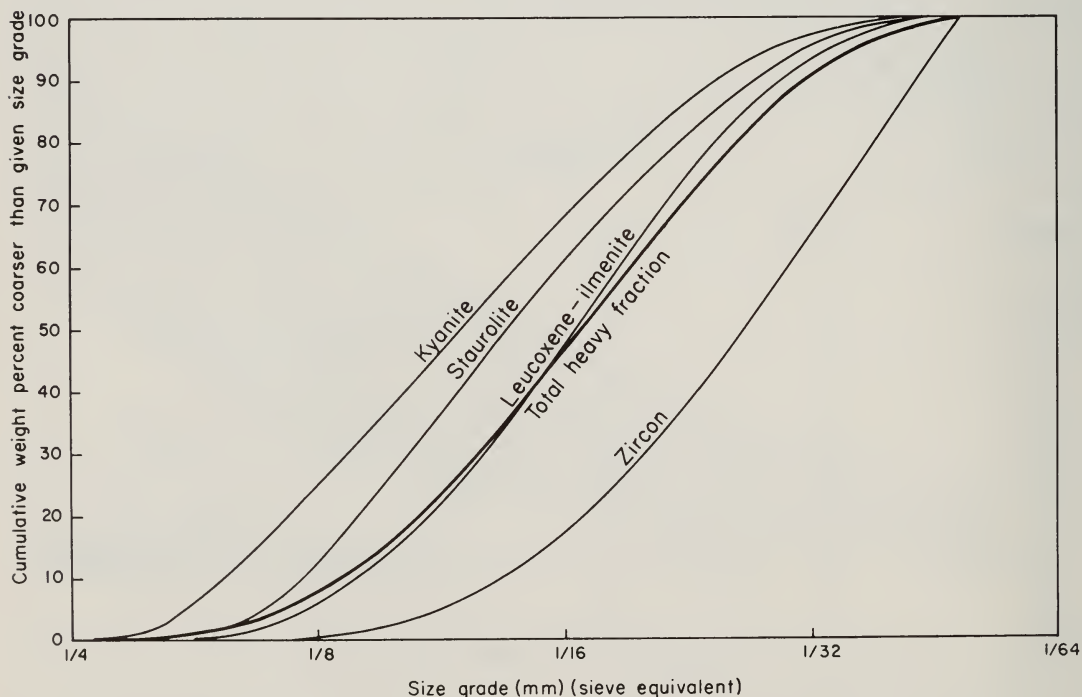


Figure 5 - Average size distributions of heavy minerals from sand of the McNairy Formation of extreme southern Illinois. The average is an unweighted average of all samples. The microscopically measured grain diameters were converted to equivalent sieve size grade by the procedure outlined by Hunter (1967).

tent of monazite plus xenotime, 1.1 percent, indicated by mineralogic analyses (table 3) of the same samples that were chemically analyzed.

#### POSSIBLE COMMERCIAL USE OF THE HEAVY MINERALS

To illustrate the factors favoring and hindering commercial exploitation of the heavy minerals in the Cretaceous and Tertiary sands of extreme southern Illinois, these sands may be compared to sands along the Atlantic Ocean and at Trail Ridge, Florida, which are currently being exploited on a large scale. Ilmenite, including ilmenite partially altered to leucoxene, is the principal mineral extracted from the Florida sands; rutile, zircon, and staurolite are also separated (Carpenter et al., 1953; Giese, Shirley, and Vallye, 1964). Monazite is not separated at Trail Ridge but is separated as a by-product from the beach sands at Jacksonville, Florida (Mertie, 1953). Methods of mineral separation used in the Trail Ridge operation include gravity separation using Humphreys Spiral Separators, electromagnetic separation, and electrostatic separation.

The heavy minerals of the southern Illinois sands are the same as those of the Trail Ridge sands, and even the proportions of the minerals are fairly similar (Spencer, 1948). Titanium minerals, for example, comprise 45 percent of the total heavy fraction of the Trail Ridge sand (Carpenter et al., 1953).

Although the mineralogy of the heavy fractions of the southern Illinois sands is strikingly similar to that of the Trail Ridge sands, the Florida sands contain larger amounts of heavy minerals. Whereas the Trail Ridge sands average about 4 per-

TABLE 5 - WEIGHT PERCENTS OF TiO<sub>2</sub>, ZrO<sub>2</sub>, Ce, La, AND Y  
IN HEAVY FRACTIONS OF SAMPLES

Sample no.	TiO <sub>2</sub>		ZrO <sub>2</sub>		Ce	La	Y
	Estimated from mineralogic analysis*	By X-ray fluo-rescence analysis**	Estimated from mineralogic analysis†	By X-ray fluo-rescence analysis**	By spectro-graphic analysis**	By spectro-graphic analysis**	By spectro-graphic analysis**
1	53		2				
2	30		3				
3	46		11				
4	37		10				
5a	38	36.8	11	7.8	0.31	0.23	0.10
5b	36	33.8	4	4.5	0.04	0.06	0.03
6	46	45.2	8	4.5	0.05	0.09	0.08
7	40	43.4	8	5.0	0.08	0.10	0.08
8	42	42.4	9	6.9	0.10	0.11	0.09
9	53	50.6	6	7.0	0.07	0.10	0.09
10a	38	36.6	2	4.5	0.03	0.03	0.02
10b	36	38.7	8	5.1	0.06	0.05	0.04
11	43	41.4	8	6.7	0.06	0.08†	0.06
12	47	41.9	10	10.7	0.20	0.23	0.20
13	45	37.8	11	8.4	0.16	0.15	0.17
14	42		8				
15a	47	41.6	7	9.0	0.11	0.12	0.11
15b	46	42.9	8	8.5	0.09	0.12	0.10

\*The estimates were made by assuming that the rutile is 100 percent TiO<sub>2</sub>, the leucoxene contains 70 percent TiO<sub>2</sub>, and the ilmenite 55 percent TiO<sub>2</sub>.

\*\*Determined by Miss Juanita Witters.

†The estimates were made by assuming that the zircon is pure ZrSiO<sub>4</sub> containing 67.2 percent ZrO<sub>2</sub>.

‡Neutron activation analysis of this sample by R. R. Ruch gave a value of 0.11 percent La.

cent heavy minerals (Carpenter et al., 1953; Giese, Shirley, and Valley, 1964), most of the sand samples from the McNairy Formation contain less than 1 percent heavy minerals, and the largest amount found was 1.4 percent (table 3). Probably considerable volumes of sand containing approximately 1 percent heavy minerals could be found by drill-hole prospecting in the McNairy Formation. Appreciably greater concentrations are unlikely, but no certain judgment can be made because of the rarity of outcrops of the McNairy Formation.

From the standpoints of percentage of heavy minerals, volume of sand available, and sparsity of interbedded clay, silt, or gravel, the other Cretaceous and Tertiary formations of extreme southern Illinois are much less promising economically than the McNairy Formation.

TABLE 6 - ESTIMATED WEIGHT PERCENTS OF RECOVERABLE TiO<sub>2</sub>, ZrO<sub>2</sub>, Ce, La, and Y IN THE SAMPLES\*

Sample no.	TiO <sub>2</sub>	ZrO <sub>2</sub>	Ce	La	Y
1	.05	.001	0	0	0
2	.06	.006	0	0	0
3	.04	.01	0	0	0
4	.04	.01	0	0	0
5a	.3	.06	0.002	0.002	0.001
5b	.1	.01	0.0001	0.0002	0.0001
6	.2	.03	0.0003	0.0005	0.0004
7	.2	.02	0.0003	0.0004	0.0003
8	.3	.04	0.0006	0.0007	0.0005
9	.6	.08	0.0008	0.001	0.001
10a	.2	.03	0.0002	0.0002	0.0001
10b	.3	.04	0.0004	0.0004	0.0003
11	.3	.05	0.0004	0.0006	0.0004
12	.3	.06	0.001	0.001	0.001
13	.5	.1	0.002	0.002	0.002
14	.3	.07	0.001	0.001	0.001
15a	.4	.08	0.001	0.001	0.001
15b	.3	.07	0.0007	0.001	0.0008

\*Estimates were made by multiplying the weight percent heavy fraction in the sample (table 3) by the weight percent of the given constituent in the heavy fraction of the sample (table 5). The weight percent of the given constituent as determined by chemical analysis was used where available; values estimated from mineralogic analyses were used otherwise.

It is uncertain whether the relatively fine grain sizes of the heavy minerals would hinder their separation by commonly used commercial processes.

A possible advantage of the McNairy sands, compared to the Florida sands, is their content of muscovite mica. Finely ground muscovite is used mainly as a filler in various products. It may be separated from quartz by flotation (Browning and Bennett, 1965; and Browning, Millsaps, and Bennett, 1965). The mica contents of samples 8 and 9 (table 3) suggest that possibly extensive deposits with considerably more than 1 percent by weight mica may occur in the McNairy Formation. Graphite is commonly associated with the muscovite but, in the samples examined in this study, is less than one-tenth as common as the muscovite.

After removal of the heavy minerals and mica, the sand of the McNairy Formation consists almost entirely of quartz. A product of very high silica content can

be produced from the sand (Ehrlinger et al., 1968). Possibly the best chance for utilization of the heavy minerals and mica is as co-products in the production of silica sand. However, a very large production of silica sand would be needed for appreciable quantities of heavy minerals to be produced.

### CONCLUSIONS

All of the Cretaceous and Tertiary sands of extreme southern Illinois contain assemblages of heavy minerals that are relatively similar to each other and to the heavy mineral suites of certain Florida sands that are being utilized commercially. The McNairy Formation is more promising than the other formations from the standpoint of possible commercial utilization of heavy minerals. Its sands contain concentrations of heavy minerals equal to or larger than those found in sands of the other formations, and it contains far larger volumes of sand and less interbedded clay, silt, or gravel than are found in the other formations.

The heavy fractions of the McNairy Formation samples have an average mineralogic composition of 60.3 percent ilmenite and leucosene, 5.5 percent rutile, 12.6 percent zircon, 1.1 percent monazite and xenotime, 6.5 percent staurolite, 8.0 percent kyanite, 1.4 percent sillimanite, and 4.5 percent tourmaline. The average contents of economically valuable chemical constituents in analyzed samples of McNairy Formation heavy fractions are 42.0 percent  $\text{TiO}_2$ , 6.9 percent  $\text{ZrO}_2$ , 0.10 percent Ce, 0.11 percent La, and 0.09 percent Y.

Although the heavy fractions of the McNairy sands are very similar mineralogically to those of the Florida sands, few of the McNairy Formation samples contain more than 1 percent heavy minerals, which is about one-fourth as much as the Florida sands contain. The percentage of  $\text{TiO}_2$  in the McNairy Formation samples averages 0.3, compared to about 1 percent in the sands of Trail Ridge, Florida (Carpenter et al., 1953). A factor that may compensate for the relatively low content of heavy minerals in the McNairy Formation sands is the relatively high muscovite mica content.

Information that must be known before the economic potential of the southern Illinois sands can be fully evaluated includes (1) the feasibility and cost of mining and mineral separation of these sands, (2) marketability of the various mineral concentrates and cost of transportation to potential consumers, and (3) heavy mineral content and extent of specific sand deposits that may be found by detailed prospecting.

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