

LIGHTWEIGHT AGGREGATE POTENTIALITIES
OF SOME INDIANA SHALES

by

HAYDN H. MURRAY and JOHN M. SMITH

Indiana Department of Conservation
GEOLOGICAL SURVEY
Report of Progress No. 12

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Harold W. Handley, Governor

DEPARTMENT OF CONSERVATION
E. Kenneth Marlin, Director

GEOLOGICAL SURVEY
Charles F. Deiss, State Geologist
Bloomington

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LIGHTWEIGHT AGGREGATE POTENTIALITIES OF SOME INDIANA SHALES

By Haydn H. Murray and John M. Smith

ABSTRACT

Laboratory tests show that some Indiana shales are potential sources of manufactured lightweight aggregate. Bloating, the process by which lightweight aggregates are manufactured, is caused by various constituents acting singly or in combination. Chemical composition and mineral composition of the shales and particle-size distribution of the mineral constituents are interrelated, and all of these factors contribute to the bloating of shales.

INTRODUCTION

Since World War II the demand for lightweight aggregates for manufacturing concrete has risen sharply. High costs of building construction have forced contractors and architects to economize wherever possible. The use of lightweight aggregates in concrete reduces the dead weight of a structure and thus less structural steel is needed for support. For example, a saving of \$180,000 on the cost for structural steel was reported in the construction of the General Petroleum Building in Los Angeles, Calif. , as a result of an expenditure of \$61,000 for lightweight concrete (Conley, Wilson, and Klinefelter, 1948, p. 6).

Ordinary concrete with sand and gravel aggregate weighs approximately 150 lb. per cubic foot, and concrete with a lightweight expanded shale aggregate weighs approximately 100 lb. per cubic foot. Concrete blocks (8 by 8 by 16 inches) containing expanded shale weigh between 21 and 28 lb. , and standard concrete blocks made of ordinary heavy aggregates weigh between 42 and 46 lb. (Cole and Zetterstrom, 1954, p. 3). In addition to their light weight, lightweight blocks have better acoustical and thermal properties than heavyweight concrete blocks. Other desirable properties of lightweight aggregate concrete are high strength, low absorption, low shrinkage, good elasticity, and durability. Therefore, these are reasons why production of concrete blocks using light-weight aggregate has increased tremendously in recent years (Cole and Zetterstrom, 1954, p. 1). In 1951 the production of lightweight aggregate concrete blocks was approximately 52 percent of the total production of concrete blocks.

Lightweight aggregate produced from expanded shales is relatively low priced, but transportation costs are a major factor in the

location of plants. In 1955 lightweight aggregate was produced by 316 plants in the United States (Pit and Quarry Publications, 1955).

PURPOSE OF INVESTIGATION

This study was made to test the potential use of some Indiana, shales as raw materials for manufacturing lightweight aggregate. Such aggregate was produced in Ohio, Illinois, and Kentucky before any interest was shown in Indiana. A new plant is now producing lightweight aggregate from shale of the Borden group near Brooklyn, Ind. , but other plants are needed to meet the increasing demand for lightweight concrete in the State. It is hoped that this report will stimulate interest in developing lightweight aggregate from shale and will enable future producers of lightweight aggregate in Indiana to evaluate better the location, thickness, extent, and physical properties of various shale formations.

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Appreciation is expressed to the many individuals and corporations who gave permission to collect samples from their properties.

LIGHTWEIGHT AGGREGATE

DEFINITION

The American Society for Testing Materials has published a standard specification governing lightweight aggregates for concrete (code no. C330-53T, 1955). This specification states that lightweight aggregate for use in concrete should consist of pumice, lava, tufa, slag, burned clay, burned shale, cinders, or other particles that are strong and durable. The unit weight of fine lightweight aggregate cannot exceed 70 lb. per cubic foot, and the unit weight of coarse lightweight aggregate cannot exceed 55 lb. per cubic foot.

TYPES OF LIGHTWEIGHT AGGREGATE

Lightweight aggregates can be classified as (1) natural aggregates, (2) byproduct aggregates, and (3) manufactured aggregates (Cole and Zetterstrom, 1954, p. 6). The principal natural light-

weight aggregates are pumice, a highly vesicular light-colored volcanic rock; scoria, a highly vesicular dark-colored volcanic rock; tuff, a volcanic rock composed largely of cemented particles of ash; and diatomaceous earth, a rock composed primarily of siliceous skeletons of microscopic single-celled plants. Other natural lightweight aggregates are perlite, a volcanic rock which can be “popped” (fractured into tiny particles) when heated properly, and vermiculite, a micaceous mineral which expands or exfoliates when heat is applied to it. The principal byproduct aggregates are slag, cinder, and sintered fly ash, and manufactured aggregates include artificially expanded shale, clay, slate, and other raw mineral materials.

This report is concerned only with expandable shales. Two general processing methods are used to expand shales. One is to prepare sized material before bloating in such a way that the bloated product will be of the required size, and the other method, which is the more commonly used, is to expand the shale and then to crush and size it.

PREVIOUS INVESTIGATIONS OF EXPANDED SHALE

In 1919 Hayde produced the first commercial lightweight aggregate by expanding shale and selling it under the trade name Haydite. Since that time new methods and processes have been developed, and at present expanded clays and shales are sold under a wide variety of trade names. Many articles describing manufacturing processes and raw materials have been published in trade journals.

Two conditions necessary for bloating have been recognized by ceramists for many years: (1) a material must produce a high-temperature glassy phase with a viscosity high enough to trap a gas, and (2) some substance that will evolve a gas at the temperature at which the glassy phase forms must be present. Ceramists have long considered that bloating of clays and shales is an adverse reaction in the ceramic process; only in recent years has this bloating characteristic been utilized to make a salable product.

Jackson (1903, p. 37-43) presented one of the first theories attempting to explain the bloating of clays and shales. He believed that ferric oxide was necessary for a clay to bloat, because oxygen would be liberated when the ferric oxide dissociated when it was heated.

Orton and Staley (1908) suggested that ferric oxide was not the only cause of bloating. They maintained that all clays should bloat at the same temperature if ferric oxide was the bloating agent and showed experimentally that clays actually bloated at many different temperatures. They further suggested that sulfur dioxide caused

bloating in burned shale.

Wilson (1927, p. 180) suggested that the evolution or expansion of gases from any or all of the following sources could cause bloating: entrapped air, steam, sulfur dioxide or trioxide, carbon dioxide or monoxide, oxygen, and hydrocarbons absorbed during firing.

Austin, Nunes, and Sullivan (1942) made quantitative determinations of the amounts of CO_2 , SO_3 , and H_2O that were given off during the bloating of clays. They also experimented to determine the effects of heating rate, air flow, and different atmospheres.

Conley, Wilson, and Klinefelter (1948, p. 10) worked on bloating properties of clay and shale as raw materials and in blended mixtures and also studied exhaustively the engineering aspects of bloated clay aggregates for concrete. They found that sulfates and carbonates produced excellent bloating in some clays and poor results in others. They concluded that by proper blending of admixtures many nonbloating clays could be made to bloat but that each clay was an individual problem.

Riley (1951, p. 121-128), by utilizing a large number of chemical analyses of bloating and nonbloating clays, was able to define the limits of bloating on a composition diagram. Those synthetic mixtures and igneous rocks which had compositions that fell within the bloating area on the diagram bloated well after the mixtures and rocks had been ground and cast into briquettes. The accessory minerals pyrite, hematite, and dolomite produced gases at temperatures high enough to cause bloating, and fine-grained portions of shale which apparently contain only illite also bloated.

Studies of lightweight aggregate potentialities have been made in other states in recent years. Plummer and Hladik (1951) reported the sampling and testing of clays and shales in Kansas; Mason (1951) discussed the testing of a few samples which appeared to have good lightweight aggregate possibilities in Oregon; Greaves-Walker, Bugg, and Hagerman (1951, p. 23) investigated the bloating characteristics of Florida clays, and Cole and Zetterstrom (1954, p. 43) investigated some clays and shales and other materials in the Dakotas to determine their suitability for lightweight aggregate. Burwell (1954) reported on lightweight aggregate from some shales in Oklahoma.

ANALYTICAL PROCEDURES

The writers attempted to collect samples representative of those geologic formations that crop out in Indiana and that contain reserves of shale or clay sufficient enough for commercial production of lightweight aggregate. Samples were obtained from rocks of the Silurian,

Devonian, Mississippian, Pennsylvanian, and Quaternary systems, but most samples came from Pennsylvanian and Mississippian rocks. Figure 1 shows the stratigraphic position and geologic age of the samples collected and studied for this report.

Lake clays of Wisconsin age were sampled in northern Indiana. The locations of all samples collected are indicated on plate 1.

SYSTEM	SERIES	ROCK UNITS SAMPLED
QUATERNARY	PLEISTOCENE	Wisconsin lacustrine clay
TERTIARY	PLIOCENE	
PENNSYLVANIAN	CONEMAUGH	Shelburn formation
	ALLEGHENY	Dugger formation
		Linton formation
	POTTSVILLE	Brazil formation
		Mansfield formation
MISSISSIPPIAN	CHESTER	Bethel formation
	MERAMEC	
	OSAGE	BORDEN GROUP Locust Point formation
	KINDERHOOK	New Providence shale
DEVONIAN	UPPER	New Albany shale
	MIDDLE	
SILURIAN	CAYUGAN	
	NIAGARAN	Mississinewa shale
	ALBION	

Figure 1. Geologic column of Indiana showing stratigraphic position and geologic age of samples studied for this report.

METHOD OF SAMPLING

The channel method was used in collecting all samples. A narrow, rather deep vertical channel was cut in the outcrop face to eliminate highly weathered surface material. A tarpaulin then was placed at the base of the channel and a continuous sample weighing approximately 50 lb. was collected. Table 1 shows the location at which each sample was collected, the stratigraphic unit from which each sample was obtained, and the thickness of each sample.

PREPARATION

Figure 2 shows the procedure that was used in studying the lightweight aggregate potentialities of the clay and shale samples. First, each sample was crushed in a No. 1 Denver Fire Clay jaw crusher. Each sample next was split in a Jones splitter. A 5-lb. portion of the split was set aside for testing, and the remainder of the split was placed in storage. The 5-lb. sample was ground in a Simpson mixer either for 10 minutes or until the particles of clay or shale would pass through a 12-mesh sieve. From the 5-lb. ground sample two 30-gram splits were made by a Jones splitter; one split was used for chemical analyses and pH determinations, and the other was used for X-ray analyses, differential thermal analysis of the clay, and petrographic analysis of the +300-mesh fraction. The remainder of the 5-lb. ground sample was used in making ten 1 by 1 by 3 inch briquettes for physical tests. Water was added until the ground material was workable and could be shaped in a steel mold.

TESTING PROCEDURE

Measurements of length, weight, and volume of the wet briquettes were made. The briquettes then were dried overnight at 110° C. in an oven, and measurements of the dried briquettes were recorded to calculate shrinkage, volume, and weight loss. The briquettes next were placed in a Cooley electric furnace in which the temperature was slowly raised to 1, 200° F. and maintained for 2 hours. During this calcining procedure, a Harper electric furnace was heated to 2, 180° F. An insulating brick wall was built inside the regular door of the Harper furnace to prevent extreme loss of heat when the briquettes were removed. In order to remove the briquettes one brick was removed from the inner wall. Two briquettes were withdrawn from the calcining furnace and placed in the Harper furnace as rapidly as possible on a silica-coated brick to

Table 1.--Location, stratigraphic position, and thickness of 29 samples studied for this report

Sample No.	County	Location	Stratigraphic Unit	Thickness (feet)
1 - - -	Lake	NE¼SW¼ sec.31, T. 66 N., R. 9 W.	Lacustrine clay of Wisconsin stage	8
2 - - -	Marshall	SE¼NW¼ sec. 9, T. 34 N., R. 3 E.	Lacustrine clay of Wisconsin stage	12
3 - - -	Jasper	NE¼SE¼ sec. 24, T. 27 N., R. 7 W.	New Albany shale	10
4 - - -	Grant	NW¼SE¼ sec. 35, T. 25 N., R. 6 E.	Mississinewa shale	12
5 - - -	Fountain	SW¼SW¼ sec. 32, T. 22 N., R. 7 W.	Borden group (undifferentiated)	30
6 - - -	Fountain	SW¼NW¼ sec. 31, T. 20 N., R. 7 W.	Brazil formation	40
7 - - -	Montgomery	SE¼SW¼ sec. 20, T. 19 N., R. 5 W.	Borden group (undifferentiated)	25
8 - - -	Vermillion	SE¼NW¼ sec. 36, T. 17 N., R. 9 W.	Staunton formation shale above Coal II	8
9 - - -	Vermillion	SE¼NE¼ sec. 15, T. 16 N., R. 9 W.	Linton formation shale above Coal IIIa	15
10 - - -	Vermillion	SW¼NE¼ sec. 33, T. 15 N., R. 9 W.	Dugger formation shale above Coal VII	35
11 - - -	Vermillion	NW¼SW¼ sec. 23, T. 14 N., R. 10 W.	Dugger formation shale above Coal VII	25
12 - - -	Parks	SW¼NW¼ sec. 29, T. 15 N., R. 8 W.	Linton formation shale above Coal IIIa	25
13 - - -	Clay	SW¼NE¼ sec. 2, T. 13 N., R. 7 W.	Brazil formation shale above Upper Block coal	12
14 - - -	Putnam	NW¼NW¼ sec. 6, T. 15 N., R. 5 W.	Mansfield formation	25
15 - - -	Putnam	Center sec. 19, T. 13 N., R. 4 W.	Mansfield formation	35
16 - - -	Vigo	SE¼NE¼ sec. 18, T. 12 N., R. 9 W.	Dugger formation shale above Coal VI	30
17 - - -	Morgan	NE¼NE¼ sec. 34, T. 13 N., R. 1 E.	New Providence shale	35
18 - - -	Morgan	NE¼NE¼ sec. 35, T. 13 N., R. 1 E.	New Providence shale	25
19 - - -	Knox	W¼ sec. 10, T. 4 N., R. 8 W.	Dugger formation shale above Coal VI	20
20 - - -	Lawrence	SE¼SE¼ sec. 29, T. 6 N., R. 2 W.	Bethel formation	15
21 - - -	Lawrence	SW¼NW¼ sec. 12, T. 3 N., R. 2 W.	Bethel formation	14
22 - - -	Jackson	NE¼NE¼ sec. 27, T. 5 N., R. 3 E.	Locust Point formation	28
23 - - -	Jennings	NE¼ sec. 34, T. 7 N., R. 8 E.	New Albany shale	30
24 - - -	Gibson	S½ sec. 23, T. 2 S., R. 9 W.	Dugger formation shale above Coal VI	25
25 - - -	Vanderburgh	SE¼NE¼ sec. 22, T. 6 S., R. 11 W.	Shelburn formation	40
26 - - -	Spencer	NE¼SE¼ sec. 20, T. 5 S., R. 5 W.	Brazil formation shale above Minshall Coal	25
27 - - -	Perry	SW¼NE¼ sec. 15, T. 6 S., R. 3 W.	Mansfield formation below Coal II	22
28 - - -	Floyd	NE¼SE¼ Clark Grant 63	New Albany shale	11
29 - - -	Clark	Center, Clark Grant 46	New Providence shale	9

ANALYTICAL PROCEDURES

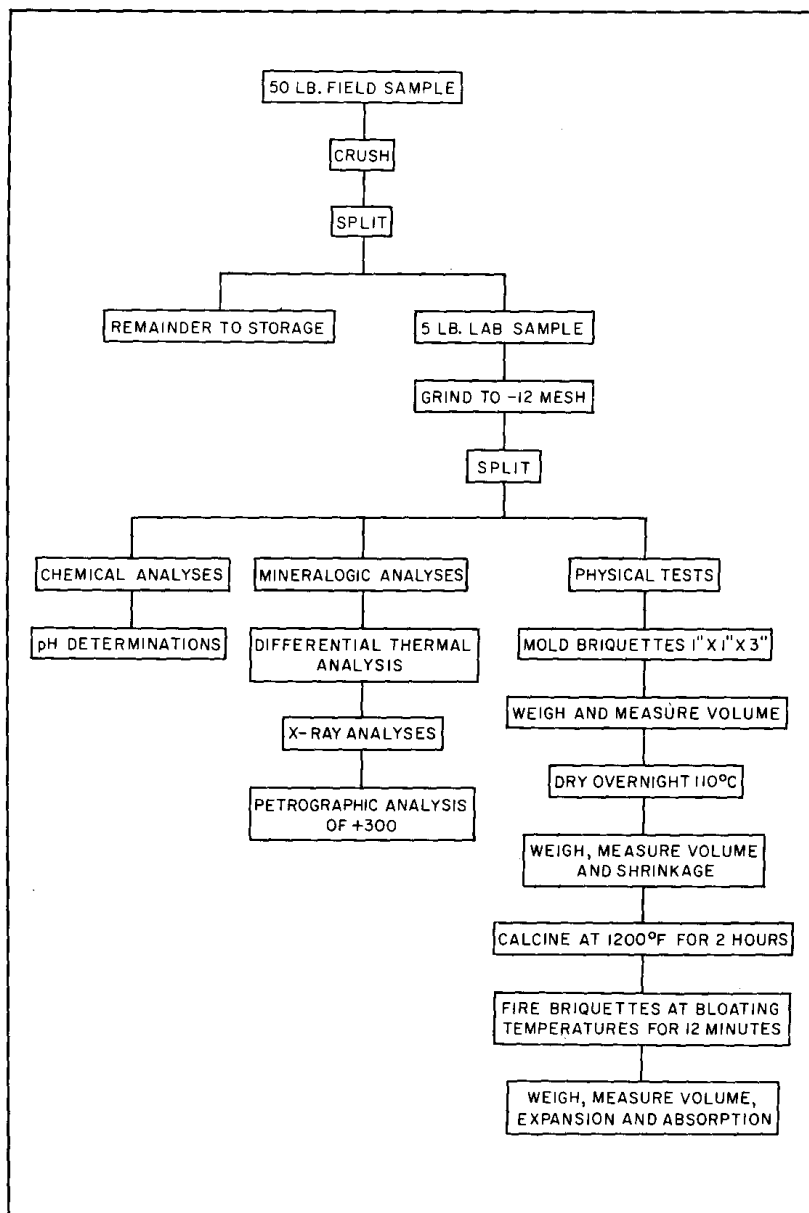


Figure 2. Flow sheet showing procedure used in analyzing samples of clay and shale for lightweight aggregate.

vent sticking. They remained in the Harper furnace for 12 minutes and then were moved and returned to the calcining (Cooley) furnace to cool.

For most samples, two bricks were placed in the bloating furnace at each of the following temperatures: 2,180° F., 2,200° F., 2,260° F., 2,300° F., and 2,340° F. The amount of expansion was observed after the first two briquettes had been fired at 2,180° F. If this temperature expanded the bricks excessively, the temperature was lowered and the next set of bricks was fired at a lower temperature. For nearly all samples 2,180° F. proved to be a satisfactory starting temperature. For a few samples, however, 2,420° F. had to be used as the minimum temperature in attempting to bloat the bricks.

After the bricks had cooled, additional physical data were obtained. The fired weight was recorded, and the water of absorption was determined by a comparison of the weight of bricks after they had been boiled in water for 3 hours with their dry-fired weight. The time of the fired bricks was determined by submerging the bricks in kerosene and measuring the volume of displaced kerosene. If the bricks were excessively porous after firing, the outside surface was coated with petroleum jelly in order to obtain a more accurate figure for the volume. Volume increase, density, water of absorption, and percentage of ignition were calculated from these measurements.

RESULTS OF BLOATING TESTS

In this report shale or clay which has a minimum volume increase ratio of 1.5 and a minimum density of 1.20 gm. per cubic centimeter or 70 lb. per cubic foot is considered bloated shale or clay. The Southeastern Experiment Station of the U. S. Bureau of Mines at Tuscaloosa, Ala., uses a temperature classification to distinguish commercial bloating from bloating (Conley, Wilson, and Klinefelter, 1948). Riley (1951, p. 121) considered any volume expansion of clay as bloating but stated that only those clays that are classified as good bloaters could be considered as possible commercial products. Most investigators agree that 2,400° F. is the maximum temperature at which a clay or shale can be fired economically to produce a bloated product.

Unbloomed vitrified shale or clay has a density of approximately 2.3 gm. per cubic centimeter or about 143 lb. per cubic foot (Plummer and Hladik, 1951, p. 28). Extreme bloating can produce a brick that has a density as low as 0.45 gm. per cubic centimeter or about 30 lb. per cubic foot.

Table 2 shows the results of bloating tests for 29 samples which the writers collected and studied. The bloating characteristics of Indiana shales are discussed below under appropriate stratigraphic headings.

SILURIAN SYSTEM

One sample (4) of Mississinewa shale from Grant County near Sweetser was tested. (See table 1.) This shale became dense and glassy as the firing temperature was increased but did not bloat. The shale at this locality is unsuitable for production of lightweight aggregate. In all probability most shales in the Mississinewa would not bloat because of the high carbonate content.

DEVONIAN SYSTEM

The major part of the New Albany shale is Devonian in age, but the upper 10 to 20 feet in southern Indiana and an undetermined thickness in northern Indiana are Mississippian in age (Campbell, 1946, p. 829). The New Albany, a fine-grained dark-gray to black shale, averages about 100 feet thick and crops out in Indiana in a belt from Clark County to Jasper County. The outcrops are numerous in the area between Clark County and Bartholomew County but are sporadic in the area from Bartholomew County to Jasper County because glacial drift mantles the bedrock.

Three samples of New Albany shale were tested for bloating characteristics. (See table 1.) Samples 3, 23, and 28 indicate that some of the New Albany shale has good bloating characteristics and would produce an aggregate of low density at relatively low temperatures. (See table 2.) Two samples (3 and 28) were exceptionally good bloaters.

MISSISSIPPIAN SYSTEM

Borden group.-- The Borden group in Indiana is composed mainly of siltstones and shales and ranges from 450 to 750 feet in thickness. In southern Indiana this group is divided, in ascending order, into five formations: New Providence shale, Locust Point formation, Carwood formation, Floyds Knob formation, and Edwardsville formation. Six samples (table 1) were collected from the Borden group.

Samples 17, 18, and 29 were collected from the New Providence shale, which is gray, fine-grained, soft, and dense. The temper

Table 2.--Results of bloating tests

Sample No.	Temperature (°F.)	Ratio of volume increase	Density		Fired absorption (pct.)	Fired apparent porosity (pct.)	Loss on ignition (pct.)
			gm. per cc.	lb. per cu. ft.			
1	1,900	0.9	1.85	115.5	12.4	22.9	9.19
	2,000	0.9	1.90	118.6	7.5	14.1	9.51
	2,100	1.3	1.33	83.0	1.8	2.4	10.5
	2,140	1.9	0.87	51.3	12.4	10.9	9.91
	2,180	1.7	0.99	61.8	10.7	10.2	9.90
2	1,900	0.9	1.59	99.3	25.0	39.3	16.6
	2,000	0.9	1.61	100.5	21.7	34.9	16.4
	2,100	0.7	2.05	128.0	1.8	3.4	17.0
	2,140	0.8	1.92	119.9	-----	-----	19.4
3	2,060	1.5	1.17	73.0	2.4	2.8	6.92
	2,100	2.3	0.74	46.2	0.2	0.2	7.71
	2,140	---	-----	-----	-----	-----	7.03
	2,240	6.9	0.24	15.0	-----	-----	9.12
4	1,900	0.9	1.53	95.5	34.0	52.2	24.8
	2,000	0.9	1.40	87.4	33.6	47.4	34.2
	2,100	0.9	1.50	93.6	31.6	14.5	25.1
	2,180	0.8	1.87	116.7	26.0	48.3	24.5
	2,220	0.7	2.17	135.5	6.8	48.3	26.1
5	2,180	1.8	1.03	64.3	3.2	3.2	5.45
	2,220	1.4	1.27	79.3	3.5	4.4	5.26
	2,260	1.8	0.98	61.2	23.6	23.3	4.98
	2,300	2.0	0.86	53.7	30.3	26.2	5.80
	2,340	2.1	0.86	53.7	19.8	17.2	5.46
6	2,180	1.4	1.23	76.8	2.2	2.6	9.45
	2,220	1.2	1.55	96.8	2.3	3.6	8.85
	2,260	1.1	1.64	102.4	0.1	2.4	9.22
	2,300	1.1	1.70	106.1	0.7	1.2	9.01
	2,340	1.1	1.59	99.3	1.1	1.8	8.84
7	2,180	1.1	1.60	99.9	0.4	0.7	3.97
	2,220	1.2	1.42	88.6	1.0	1.4	4.43
	2,260	1.7	1.02	63.7	0.7	0.7	4.23
	2,300	1.8	0.93	58.1	1.1	1.0	4.20
	2,340	2.0	0.85	53.1	3.4	3.0	4.20
8	2,180	0.8	2.36	147.3	-----	-----	5.05
	2,300	1.1	1.60	99.9	-----	-----	3.65
	2,400	0.7	1.43	89.3	-----	-----	5.23
9	2,180	---	-----	-----	13.9	-----	12.3
	2,220	1.8	0.86	53.7	37.0	12.0	12.4
	2,260	2.3	0.69	43.1	1.7	25.4	12.4
	2,300	2.8	0.57	35.6	4.4	2.5	12.3
	2,340	2.6	0.61	38.1	5.3	3.4	12.0
10	2,180	1.5	1.12	69.9	3.1	3.4	9.11
	2,300	1.6	1.02	63.7	2.1	2.1	8.07
	2,360	1.8	0.91	56.8	4.5	4.0	7.58
	2,400	2.3	0.73	45.6	17.5	12.8	7.49

Table 2.--Results of bloating tests--Continued

Sample No.	Temperature (°F.)	Ratio of volume increase	Density		Fired absorption (pct.)	Fired apparent porosity (pct.)	Loss or ignition (pct.)
			gm. per cc.	lb. per cu. ft.			
11	2,180	1.3	1.25	78.0	2.2	3.5	6.67
	2,220	1.6	1.10	68.7	2.6	2.9	7.97
	2,280	2.3	0.75	46.8	7.4	5.6	6.47
	2,300	3.1	0.53	33.1	3.5	1.9	6.90
	2,340	4.1	0.43	26.8	41.5	17.6	6.93
12	2,180	1.7	1.01	63.0	2.4	2.4	7.07
	2,220	1.0	1.54	96.1	----	----	6.36
	2,260	1.0	1.67	104.3	1.4	----	8.14
	2,300	1.2	1.48	92.4	0.8	1.1	6.72
	2,340	1.3	1.40	87.4	1.6	----	6.65
13	2,180	1.2	1.48	92.4	4.6	6.7	8.28
	2,220	1.0	1.67	124.3	3.4	5.6	7.87
	2,300	1.1	1.53	95.5	3.1	4.7	7.70
	2,360	1.4	1.21	75.5	1.2	1.5	7.75
	2,400	1.3	1.25	78.0	2.2	2.8	8.07
14	2,180	0.9	1.84	114.9	0.6	1.1	7.76
	2,260	1.1	1.70	106.1	0.1	0.2	7.92
	2,360	1.3	1.43	89.3	0.8	1.2	8.11
	2,400	1.2	1.47	91.8	1.3	1.9	7.89
15	2,180	1.0	1.70	106.1	2.2	3.7	9.25
	2,220	1.1	1.58	98.6	2.0	3.2	9.19
	2,260	1.2	1.41	88.0	1.8	2.4	9.18
	2,300	1.2	1.33	83.0	1.9	2.4	9.10
	2,360	1.6	1.05	65.5	1.7	1.8	9.43
16	2,180	0.8	2.20	137.3	0.1	0.3	5.08
	2,260	1.2	1.47	91.8	0.6	1.0	4.90
	2,300	1.2	1.45	90.5	0.6	0.9	4.84
	2,340	1.5	1.22	76.2	1.1	1.3	5.10
	2,400	1.8	1.05	65.5	4.0	4.3	5.64
17	2,180	---	----	-----	1.0	----	4.60
	2,300	1.9	0.88	54.9	0.7	0.6	4.20
	2,340	2.2	0.77	48.1	16.6	12.9	4.28
	2,380	2.7	0.62	38.7	14.8	9.1	4.52
	2,420	2.9	0.57	35.6	24.7	13.8	4.89
18	2,180	1.7	0.98	61.2	0.9	0.9	6.83
	2,220	2.2	0.75	46.8	0.9	0.6	7.16
	2,260	2.7	0.60	37.5	1.8	1.1	5.80
	2,300	3.2	0.50	31.2	11.2	5.6	7.22
	2,340	3.2	0.50	31.2	25.8	12.6	7.79
19	2,180	1.1	1.57	98.0	----	----	7.26
	2,220	1.2	1.47	91.8	----	----	7.31
	2,260	1.4	1.22	76.2	1.2	1.6	7.51
	2,300	1.7	1.03	64.3	2.5	2.6	7.59
	2,340	1.9	0.93	58.1	4.2	3.8	7.33
20	2,100	1.8	1.05	65.5	7.0	6.7	6.96
	2,180	2.5	0.72	44.9	9.4	7.5	7.48
	2,220	3.0	0.60	37.5	9.8	5.9	8.09
	2,260	5.3	0.34	21.2	19.4	6.6	7.90
	2,300	7.0	0.25	15.6	28.6	7.2	8.39

Table 2.--Results of bloating tests--Continued

Sample No.	Temperature (°F.)	Ratio of volume increase	Density		Fired absorption (pct.)	Fired apparent porosity (pct.)	Loss on ignition (pct.)
			gm. per cc.	lb. per cu. ft.			
21	2,180	1.6	1.14	71.2	5.8	6.6	7.79
	2,260	2.2	0.80	49.9	-----	-----	8.23
	2,300	2.8	0.62	38.7	23.9	14.5	8.14
	2,340	4.1	0.44	27.2	14.2	5.6	8.82
	2,360	5.1	0.34	21.5	21.4	7.7	10.3
22	2,180	1.2	1.38	86.1	1.0	1.4	5.20
	2,220	1.6	1.07	66.8	1.8	1.9	5.00
	2,260	1.8	0.91	56.8	2.2	2.0	5.51
	2,300	1.9	0.85	53.1	4.5	3.8	4.99
23	2,180	1.7	1.03	64.3	24.9	25.3	12.3
	2,200	1.6	1.05	65.5	27.5	24.2	13.9
	2,220	2.2	0.76	47.4	32.2	31.9	12.7
	2,260	2.3	0.73	45.6	44.2	21.6	13.3
24	2,180	1.1	1.57	98.0	0.2	0.2	6.73
	2,220	1.4	1.24	77.4	0.2	0.2	7.24
	2,260	1.5	1.17	73.0	1.7	2.0	6.82
	2,300	1.6	1.08	67.4	1.0	1.2	7.48
	2,340	1.9	0.91	56.8	3.8	3.4	6.72
25	2,180	0.8	2.17	135.5	-----	-----	5.12
	2,220	1.1	1.63	101.8	-----	-----	5.76
	2,260	1.3	1.37	85.5	0.6	0.9	5.64
	2,300	1.4	1.26	78.7	1.8	2.4	5.53
	2,340	1.7	1.05	65.5	6.1	6.4	5.63
26	2,180	1.4	1.20	74.9	5.2	6.2	7.40
	2,220	1.2	1.46	91.1	3.3	4.8	7.21
	2,260	1.2	1.38	86.1	3.2	4.4	7.43
	2,300	1.4	1.19	74.3	11.2	13.0	7.42
	2,340	1.6	1.09	68.0	14.4	15.8	6.98
27	2,180	0.9	1.82	113.6	1.2	2.2	8.29
	2,220	0.7	2.26	141.1	0.2	0.4	7.76
	2,260	0.7	2.16	134.8	-----	-----	7.98
	2,300	0.9	1.81	113.0	0.2	0.3	7.79
	2,400	1.1	1.59	99.3	0.2	0.2	7.81
28	2,140	1.3	1.29	80.5	13.4	17.6	11.5
	2,180	1.7	1.00	62.4	10.2	10.4	10.9
	2,220	2.4	0.67	41.8	11.6	7.8	11.6
	2,260	3.2	0.52	32.5	13.4	6.9	12.0
	2,300	4.0	0.41	25.6	15.4	6.1	12.1
29	2,180	1.9	0.84	52.4	1.8	1.5	4.9
	2,220	2.2	0.73	45.6	2.8	2.4	4.8
	2,260	2.6	0.74	46.2	3.0	2.2	5.0
	2,300	2.3	0.71	44.3	3.8	2.7	5.0
	2,340	2.3	0.71	44.3	4.0	2.8	5.3

atures and densities of bloating for the New Providence shale were slightly higher than those for the New Albany shale, but all three samples gave results (table 2) which indicate that the New Providence shale has good bloating characteristics. One sample (18), an exceptionally good bloater, is similar to the shale which now is being used by Midwest Aggregates, Inc. , at Brooklyn, Ind., for producing lightweight aggregate.

A single sample (22) was collected for bloating tests from the Locust Point formation, which is massive, bluish gray, hard, and silty. The sample bloated and would produce lightweight aggregate; both absorption and porosity of this sample were rather low.

The Borden group has not been differentiated into formations in the northern outcrop area from which two samples (5 and 7) were collected. Both samples bloated and would produce lightweight aggregate. Absorption and porosity of the two samples were distinctly different; they were rather high for sample 5 and low for sample 7.

Bethel formation.--The Bethel formation, formerly known as the Mooretown sandstone, consists of dark-gray shales and argillaceous sandstones 5 to 30 feet thick. The formation crops out from Owen County on the north to Harrison County on the Ohio River. Two samples (20 and 21) were collected for this study. Both of them bloated exceptionally well (table 2), but their absorption and porosity were rather high. Further detailed studies of these shales should be made because they show promise of making good lightweight aggregate.

PENNSYLVANIAN SYSTEM

Mansfield formation.--The Mansfield formation, consisting of sandstone, shale, coal, underclay, limestone, and conglomerate, crops out from Warren County southeastward to Perry County on the Ohio River. The shales collected for this study (samples 14, 15, and 27) were fine-grained and massive. (See table 1.) The three samples did not show promise of making suitable lightweight aggregate. (See table 2.) Sample 15 bloated very slightly at 2,380 ° F. , which probably is too high a temperature for commercial production.

Brazil formation.--The Brazil formation contains sandstone, shale, underclay, limestone, and coal (including Lower Block coal, Upper Block coal, Minshall coal, and Coal II). This formation crops out along the west edge of the exposure area of the Mansfield. Three shale samples (6, 13, and 26) (table 1) were collected from the Brazil formation. One Sample (26) bloated at 2,340° F. (table 2), and the other samples (6 and 13) did not bloat. Inasmuch as the porosity and permeability of the bloated samples were high, the

shales tested from the Brazil formation did not show much promise as a source for lightweight aggregate.

Staunton formation.--The Staunton formation, which contains the same rock types as the Brazil formation and also includes Coal III, crops out from Vermillion County southeastward to Spencer County on the Ohio River. The sample (8) (table 1) collected from this formation did not bloat. (See table 2.) As the Staunton formation contains many other shales, one test does not preclude the possibility that other shales in the Staunton may make suitable lightweight aggregate.

Linton formation.--The Linton formation, which contains the same general lithologies as described above in the Brazil formation and also includes Coals IIIa and IV, crops out from Vermillion County to Spencer and Warrick Counties. Two shale samples (9 and 12) (table 1) were collected from the formation. Sample 9 from Vermillion County showed good bloating characteristics, but sample 12 from Parke County did not bloat. (See table 2.)

Dugger formation.--The Dugger formation, which contains sandstone, shale, underclay, limestone, and two commercial coals (Coals VI and VII), crops out from Vermillion County southeastward to Warrick County. Five shale samples (10, 11, 16, 19, and 24) (table 1) were collected from the Dugger formation for testing. All five samples bloated; sample 11 from Vermillion County gave the best results. (See table 2.)

Shelburn formation.--The Shelburn formation, consisting of shale, sandstone, underclay, thin coal, and limestone, crops out from Vigo County southward to Vanderburgh and Posey Counties. The only sample (25) collected from this formation (table 1) bloated moderately. (See table 2.)

QUATERNARY SYSTEM

Two samples (no. 1 from Lake County and no. 2 from Marshall County) of lake clays of the Wisconsin stage were collected in northern Indiana. Sample 1 bloated moderately (table 2), but sample 2 shrank and became denser at high temperatures. The use of Pleistocene lake clays would require extreme care because their compositions vary within short distances.

CHEMICAL COMPOSITION OF SAMPLES

A close relationship exists between the chemical composition and the bloating characteristics of clay and shale. In his study of this relationship of chemical properties to bloating, Riley (1951, p. 121-128) concluded that the viscosity of the melt produced by firing is determined essentially by the bulk chemical composition based upon SiO_2 , Al_2O_3 , and the total of CaO , MgO , FeO , Fe_2O_3 , and $(\text{K}, \text{Na})_2\text{O}$ in which optimum viscosity of the melt might be expected. Figure 3 shows the composition of the shales used for the present study plotted according to Riley's method. All the shales except samples 2 and 4 fall within the range of chemical composition which should produce a mass of the proper viscosity at the bloating temperature. That all the shales did not bloat indicates that some

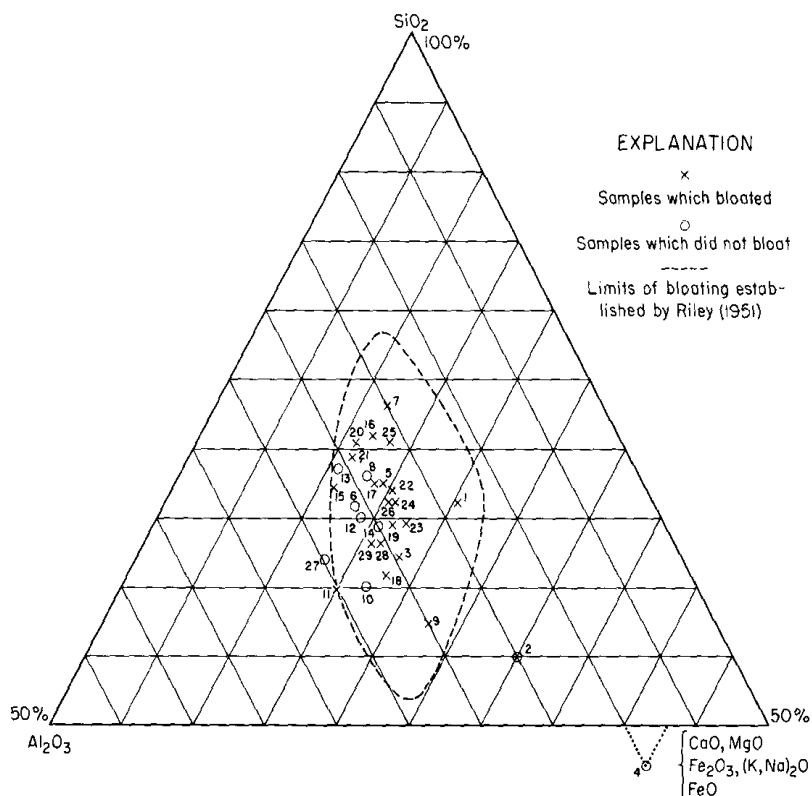
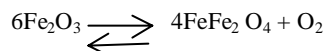


Figure 3. Composition diagram of shales plotted according to method used by Riley, 1951.

probably did not contain a constituent which would produce a gas at the proper temperature and that particle-size distribution and mineralogy are important factors in bloating.

Conley, Wilson, and Klinefelter (1948) tabulated 80 analyses of various types of clays and shales and observed (p. 16) that "the difficulty in correlations of bloating properties with the chemical analyses do not reveal the mineral form in which the constituent is present. Data from other sources... all point to the mineral form of the impurities as being the real key. With such information, the chemical analyses would then be a decided help." Conley and his associates also showed that pH is a good criterion for identifying shales that will bloat. Those that did not bloat produced a water slurry with pH less than 5, and those that bloated produced a water slurry with pH greater than 5. All shales examined in this study had a pH greater than 5 (table 3), and there appeared to be little or no relationship between pH and bloating ability in the pH range above 5.

Austen, Nunes, and Sullivan (1942) pointed out that H_2O , CO_2 , and SO_3 , were evolved as gases from several bloating clays. Mielenz and King (1955, p. 243) stated that CO , O_2 , and possibly H_2 were involved in vesiculation in many clays and shales. Riley (1951, p. 127) believed that the most significant reaction involved in generating a gas is partial reduction of ferric oxide:



At least 7.6 percent by weight of Fe_2O_3 must be present for adequate vesiculation if this source alone furnished gas. Chemical analyses of the samples studied for this report (table 4) show that the Fe_2O_3 content is not as high as Riley believed necessary; therefore, in many samples which bloated, gases also must have been produced from other sources.

Composition diagrams for all samples were plotted using SiO_2 - Al_2O_3 - S, SiO_2 - Al_2O_3 - Ignition loss, SiO_2 - Al_2O_3 - CO_2 , SiO_2 - Al_2O_3 - MgO , SiO_2 - Al_2O_3 - CaO , SiO_2 - Al_2O_3 - Fe_2O_3 , SiO_2 - Fe_2O_3 - S, SiO_2 - Fe_2O_3 - Ignition loss, SiO_2 - Fe_2O_3 - CO_2 , SiO_2 - Fe_2O_3 - MgO , and SiO_2 - Fe_2O_3 - CaO . Each 3-component entity was recalculated to 100 percent. No significant relationship could be shown between any of the three component groups listed above and the bloating characteristics. Figure 4 is a composition diagram of SiO_2 - Al_2O_3 - Ignition loss for all samples studied for this report. The factors which cause bloating are more complicated than just the chemical composition alone, and certainly more than three components enter into the chemical reaction that takes place during bloating.

In addition to the reduction of ferric oxide, gases may be pro-

Sample No.	pH
1 - - - - -	7.98
2 - - - - -	8.38
3 - - - - -	7.51
4 - - - - -	8.22
5 - - - - -	7.72
6 - - - - -	7.58
7 - - - - -	7.38
8 - - - - -	7.88
9 - - - - -	7.32
10 - - - - -	7.18
11 - - - - -	7.94
12 - - - - -	8.02
13 - - - - -	7.21
14 - - - - -	7.55
15 - - - - -	5.19
16 - - - - -	9.20
17 - - - - -	6.91
18 - - - - -	6.99
19 - - - - -	8.95
20 - - - - -	7.58
21 - - - - -	7.88
22 - - - - -	6.98
23 - - - - -	7.53
24 - - - - -	7.85
25 - - - - -	8.58
26 - - - - -	7.40
27 - - - - -	6.39
28 - - - - -	7.69
29 - - - - -	6.92

Table 4.--*Chemical analyses of samples*
[Maynard E. Collor and R. K. Leininger, Analysts]

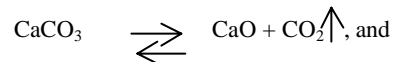
Sample No.	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	S	CO ₂	H ₂ O(+)	H ₂ O(+)	Ignition loss	Total (percent) ¹
1	57.7	12.3	5.18	0.52	4.90	3.65	0.70	3.49	0.053	0.081	0.28	5.63	1.56	3.43	10.9	99.47
2	44.3	12.2	4.46	0.49	12.7	3.98	0.64	2.89	0.064	0.11	0.029	12.8	2.28	3.39	18.5	100.33
3	56.0	17.5	5.58	0.67	1.43	3.13	0.13	6.00	0.067	0.032	0.88	1.85	1.35	4.51	8.59	99.13
4	34.0	7.43	2.06	0.30	16.5	10.8	0.22	3.91	0.023	0.055	0.12	23.9	0.37	0.71	25.1	100.40
5	63.6	17.0	5.69	0.78	0.92	2.21	0.93	4.30	0.039	0.10	0.20	0.83	0.90	3.07	5.00	100.57
6	59.2	18.8	6.60	0.80	*	1.72	0.15	3.10	0.13	0.14	0.041	2.48	1.30	5.58	9.40	100.04
7	69.6	13.8	5.20	0.80	0.34	2.02	1.39	3.10	0.026	0.11	0.27	0.48	0.53	2.49	3.77	100.16
8	62.8	17.7	5.18	0.84	0.55	2.03	1.13	3.35	0.052	0.14	0.014	0.61	0.88	4.20	5.70	99.48
9	49.3	19.9	9.81	0.65	1.10	2.37	0.67	3.48	0.13	0.36	0.80	3.56	1.21	6.83	12.4	100.17
10	54.4	21.0	7.14	0.75	0.85	2.70	0.78	3.74	0.096	0.17	0.33	1.49	1.48	5.16	8.46	100.09
11	55.0	22.0	6.36	0.65	0.56	2.69	0.98	3.74	0.084	0.15	0.40	1.54	1.41	4.66	8.01	100.22
12	59.2	19.6	6.26	0.83	0.56	2.27	1.00	3.02	0.075	0.15	0.10	1.87	0.75	4.12	6.84	99.80
13	61.4	18.8	5.48	0.78	0.18	1.52	0.23	3.00	0.12	0.092	0.37	1.54	1.20	5.33	8.44	100.04
14	57.6	18.8	7.47	0.75	0.65	2.23	0.52	3.49	0.18	0.19	0.023	2.07	0.96	5.09	8.14	100.02
15	59.8	19.2	5.55	0.80	0.27	1.23	0.17	2.66	0.069	0.087	0.44	1.60	1.78	6.58	10.4	100.24
16	66.5	16.2	4.60	0.80	0.54	2.08	1.54	2.57	0.074	0.13	0.025	1.59	0.63	3.11	5.36	100.39
17	64.0	17.1	5.97	0.77	0.38	2.47	1.22	3.70	0.037	0.093	0.33	0.34	0.70	3.37	4.74	100.48
18	56.3	19.2	7.05	0.73	2.13	2.38	0.88	4.32	0.066	0.096	0.035	2.05	1.00	3.75	6.84	99.99
19	58.9	17.6	7.21	0.67	1.48	2.00	0.97	3.38	0.14	0.18	0.095	3.80	0.77	3.09	7.76	100.29
20	63.0	16.2	4.46	0.67	1.39	1.97	0.14	2.70	0.016	0.082	1.3	0.57	3.77	4.20	9.84	100.47
21	61.8	16.7	4.27	0.79	1.76	1.67	0.10	2.56	0.017	0.084	1.6	1.13	2.76	4.30	9.79	99.54
22	63.2	16.4	7.26	0.84	0.13	2.20	1.10	4.21	0.065	0.11	0.16	0.90	0.95	3.46	5.47	100.99
23	56.6	15.3	4.10	0.63	4.24	2.90	0.27	4.00	0.096	0.055	1.2	5.84	1.03	3.36	11.4	99.62
24	60.8	17.2	6.75	0.76	0.47	2.34	1.08	3.08	0.14	0.17	0.063	2.41	0.90	4.33	7.70	100.49
25	65.8	15.3	5.74	0.77	0.67	2.04	1.26	2.66	0.074	0.14	0.055	1.80	0.64	2.97	5.57	100.02
26	60.3	17.0	7.32	0.75	0.63	2.10	0.97	2.90	0.078	0.16	0.36	1.80	0.56	4.69	7.41	99.62
27	56.9	23.1	6.58	0.84	<0.05	1.22	0.19	2.99	0.038	0.10	0.019	0.06	1.94	6.85	8.87	100.88
28	55.0	17.3	5.55	0.62	1.50	2.16	0.54	4.37	0.057	0.058	2.0	2.74	0.93	6.97	12.6	99.80
29	58.8	19.2	7.53	0.75	<0.05	2.45	0.76	4.62	0.061	0.062	0.12	0.17	1.50	4.23	6.02	100.30

¹Percentage of ignition loss is not included in total percentage.

*Not detected

CHEMICAL COMPOSITION OF SAMPLES

duced by the oxidation of pyrite with the release of SO_2 - $4\text{FeS}_2 + 11\text{O}_2 \rightarrow 2\text{Fe}_2\text{O}_3 + 8\text{SO}_2$, \uparrow by the decomposition of calcite or dolomite to CO_2 , \uparrow



by the liberation of hydroxyls from clay minerals, which in turn would release $\text{O}_2 + \text{H}_2$ as gases. Any one of these reactions or a combination of them could produce a gas that would bloat the material if the viscosity were high enough to trap the gas. Analyses also show that too much CaCO_3 or $\text{CaMg}(\text{CO}_3)_2$ causes fluxing and prevents bloating. Liberation of too much gaseous material would not produce a sound aggregate. In all probability a minimum and maximum percentage for certain constituents could be established; outside this percentage range bloating would not take place.

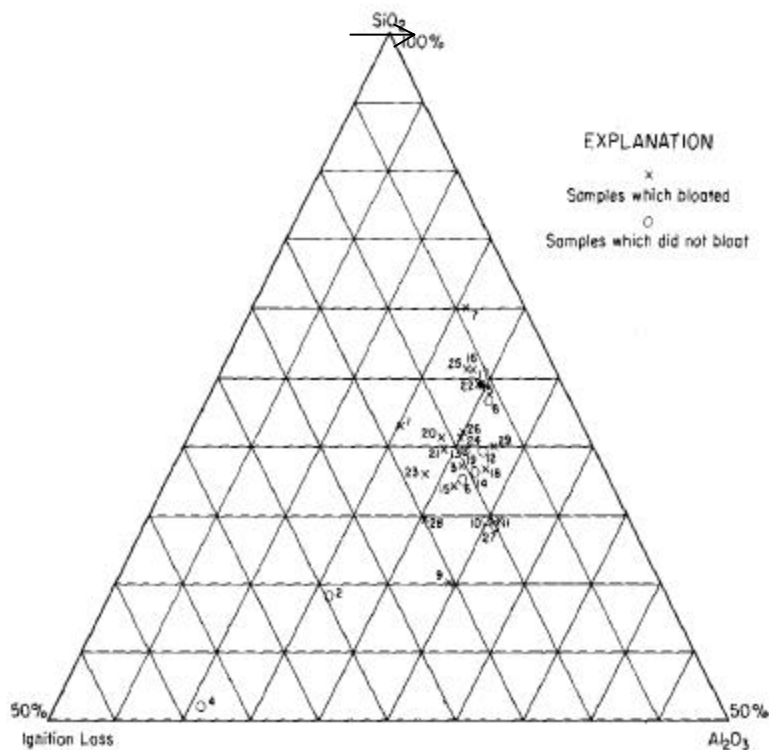


Figure 4. Composition diagram of SiO_2 - Al_2O_3 -ignition loss based on samples studied for this report.

PARTICLE-SIZE DISTRIBUTION OF SAMPLES

Mielenz and King (1955, p. 245) suggested that a dense, rather impervious clay that resists shrinkage during heating and inhibits the release of vapors and gases before fusion is the most suitable type of shale or clay for lightweight aggregate. Porous and permeable materials permit rapid loss of vapors and gases and generally bloat poorly. Although the fabric of the clays used in the present investigation was not studied, the percentage of sand, silt, and clay for each shale sample was determined by the hydrometer method described by Buoyoucos (1928, p. 365) and is shown in table 5.

No apparent correlation between bloating characteristics and the amount of sand, silt, and clay in each sample studied was discovered. The relationship between fabric and bloating should be investigated because particle orientation and arrangement undoubtedly are important to the fired strength and vesiculation of the aggregate particles. Particle-size data suggest that fabric is more important than differences in the size gradation especially when the size range of most of the samples falls within the silt and clay size.

MINERAL COMPOSITION OF SAMPLES

Minerals in the shale samples were identified with the aid of X-ray diffractometer analyses, differential thermal analyses, and petrographic analyses. A part of each sample was sieved wet through an ASTM 300-mesh sieve, and the mineral grains retained on the sieve were identified with a petrographic microscope by Wayne M. Bundy, of the Industrial Minerals Section, Indiana Geological Survey. Another part of each shale sample, crushed to pass a 200-mesh sieve, was analyzed by X-ray powder diffraction methods. A third part of each shale sample was disaggregated in distilled water, and the less-than-2-micron-fraction was separated by a sedimentation procedure based on Stokes' law of settling velocity. The less-than-2-micron-fraction is composed principally of clay minerals, which were identified by X-ray powder diffraction methods (Murray, 1954, p. 57-60). A part of the less-than-2-micron-fraction was used for differential thermal analyses.

Table 6 shows the relative abundance of minerals retained on the 300-mesh sieve. Quartz, the most abundant mineral constituent of the sand-size portion of the shales, occurs as subrounded to subangular equidimensional grains. In many shales pyrite replaces the quartz. Like quartz, calcite and dolomite, which were moderately abundant in many of the samples as microcrystalline aggregates and rhombic cleavage fragments, are partly replaced by pyrite.

Table 5: -Percentage of sand, silt, and clay in samples

Sample No.	Percent by weight		
	Sand X 047 mm.	Silt > .0042 < .047 mm.	Clay < .0042 mm.
1 - - - - -	18.4	54.4	27.2
2 - - - - -	6.4	39.2	54.4
3 - - - - -	39.8	36.8	23.4
4 - - - - -	20.8	55.6	23.6
5 - - - - -	29.9	47.4	22.7
6 - - - - -	37.4	41.2	21.4
7 - - - - -	13.4	68.2	18.4
8 - - - - -	17.4	59.0	23.6
9 - - - - -	33.5	37.3	29.2
10 - - - - -	38.4	41.4	20.2
11 - - - - -	26.7	38.2	35.1
12 - - - - -	18.0	52.6	29.4
13 - - - - -	18.2	42.5	39.3
14 - - - - -	35.4	41.6	23.0
15 - - - - -	30.0	42.7	27.3
16 - - - - -	16.8	58.0	25.2
17 - - - - -	16.5	61.8	21.7
18 - - - - -	28.2	43.0	28.8
19 - - - - -	9.0	33.2	57.8
20 - - - - -	15.8	41.8	42.4
21 - - - - -	49.7	34.7	15.6
22 - - - - -	11.4	61.4	27.2
23 - - - - -	44.6	32.8	22.6
24 - - - - -	15.6	50.2	34.2
25 - - - - -	17.4	58.2	24.4
26 - - - - -	18.0	57.8	24.2
27 - - - - -	8.6	50.6	40.8
28 - - - - -	48.4	12.8	38.8
29 - - - - -	4.2	50.2	45.6

Table 6: .Relative abundance of minerals retained on 300-mesh sieve

Sample No.	Quartz	Calcite Dolo-mite	Pyrite	Magnet-ite	Coe-hire	Leucox-ene	Biotile	Zircon	Tourma-line	Pyrox-ene	Plagi-oclase	Potash Feld-spar	Chlo-rite	Musco-vite	Horn-blends	Epidote	Garnet	Corun-dum
1	F	B	--	A	A	A	B	--	--	--	A	B	--	--	A	--	--	--
2	E	C	--	B	A	A	A	--	A	--	A	B	--	--	A	B	--	--
3	A	A	--	--	E ¹	E ¹	A	--	--	--	--	--	--	--	--	--	--	--
4	C	E	--	--	A	A	--	--	--	--	--	A	--	A	--	--	--	--
5	F	B	A	A	A	A	A	--	--	--	A	--	--	A	--	--	--	--
6	F	--	--	B	A	A	A	--	--	--	--	A	--	A	--	--	--	--
7	E	B	A	--	A	A	B	--	--	--	A	B	--	A	--	--	--	--
8	E	--	--	A	A	A	B	--	--	--	A	A	--	C	--	--	--	A
9	C	B	B	D	A	A	A	--	--	--	--	--	--	B	--	--	A	--
10	D	D	D	C	A	A	A	--	--	--	--	--	--	--	--	--	--	--
11	E	B	C	A	A	A	A	--	--	--	A	A	--	--	--	--	--	--
12	E	A	B	A	A	A	A	--	--	--	A	A	A	B	--	--	--	--
13	F	--	B	A	A	A	A	A	A	B	--	A	--	B	--	--	--	--
14	E	--	--	D	A	A	B	--	--	--	--	--	--	--	--	--	--	--
15	F	--	B	A	A	A	A	--	--	--	--	A	--	B	--	--	--	--
16	F	--	B	A	A	A	A	--	--	--	--	--	A	B	--	--	--	--
17	D	--	E	B	A	A	A	--	--	--	--	--	--	A	--	--	--	--
18	C	D	D	A	A	A	A	--	--	--	--	--	--	--	--	--	--	--
19	E	C	--	A	A	A	A	--	--	--	A	A	--	C	--	--	--	--
20	F	A	C	A	A	A	A	--	--	--	A	A	--	--	--	--	--	--
21	F	B	C	A	A	A	A	--	--	--	B	--	--	--	--	--	--	--
22	D	--	C	B	A	A	C	--	--	--	--	A	B	A	--	--	--	--
23	D	B	B	A	A	A	C	--	--	--	--	--	B	--	--	--	--	--
24	F	--	--	A	A	A	A	--	--	--	A	A	--	B	--	--	--	--
25	F	A	A	A	A	A	A	--	--	--	--	A	A	B	--	--	--	--
26	E	--	A	A	A	A	A	--	A	--	A	A	--	B	--	--	--	--
27	C	--	--	A	A	A	D	--	--	--	A	A	--	A	--	--	--	--
28	C	--	B	C	A	A	D	--	--	--	--	--	--	--	--	--	--	--
29	C	--	C	D	A	A	B	--	--	--	--	--	--	--	--	--	--	--

Percentage of grains retained on 300-mesh sieve

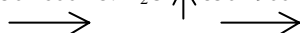
A - 0-5 percent; H - 5-10 percent; C - 10-25 percent; D - 25-50 percent; E - 50-75 percent; F - 75-100 percent

¹Almost all grains were heavily coated with goethite and leucoxene and were not identifiable.

K-Feldspar 1,050° C. Leucite and cristobalite



Chlorite 550°-600° C. H₂O ↑ 850°-900° C. Olivine



Illite 600° C. H₂O ↑ 850° C. Spinel



Calcite 850° C. CaO+CO₂ ↑



Dolomite 750° C. CaCO₃+Periclase+CO₂ ↑ 850° C. CaO+CO₂ ↑



Pyrite 450° C. Fe₂O₃+ SO₂ ↑



When rapid vitrification of the outer surface of a shale particle takes place, the rather impermeable, vitrified plastic sheath contains the volatiles and thus expands. The minerals which can release volatiles and thus contribute to bloating are kaolinite, chlorite, illite, calcite, dolomite, and pyrite. In addition, iron oxide and organic material which is considered noncrystalline can contribute to bloating.

As organic material and amounts of fine-grained calcite and dolomite less than 5 percent are not revealed by X-ray diffraction, differential thermal analysis was used for complete identification of minerals. Differential thermal analysis curves (fig. 7) could not be used for complete identification of clay minerals because differential thermal analysis does not reveal fully the clay minerals present in most of the samples. Figure 7 shows that sample 24 contained a considerable amount of organic material, which caused a broad exothermic bulge between 300° C. and 500° C.; sample 21 contained fine-grained calcite, which caused an endothermic peak at approximately 850° C.; and sample 13 contained illite, chlorite, and kaolinite in the clay-size portion, but the curve does not reveal the presence of either illite or chlorite.

Sulfur appears to be associated with organic material either in pyrite or in organic molecules, as all samples but one (18) that contained rather abundant amounts of organic material showed high sulfur percentages in the chemical analyses and bloated well.

A study of the six samples that bloated best (3, 11, 18, 20, 21, and 28) showed that any one mineral or a combination of several minerals could cause bloating. Sample 21 had an abnormally high percentage of mixed layer illite and chlorite, some calcite in the clay-size portion of the sample, a moderate amount of organic material, and a high sulfur content. Sample 18 was shale that had a high illite content, some fine-grained calcite, and a moderate amount of organic material. Sample 11 had a high percentage of clay minerals, a moderate amount of organic material, and a rather

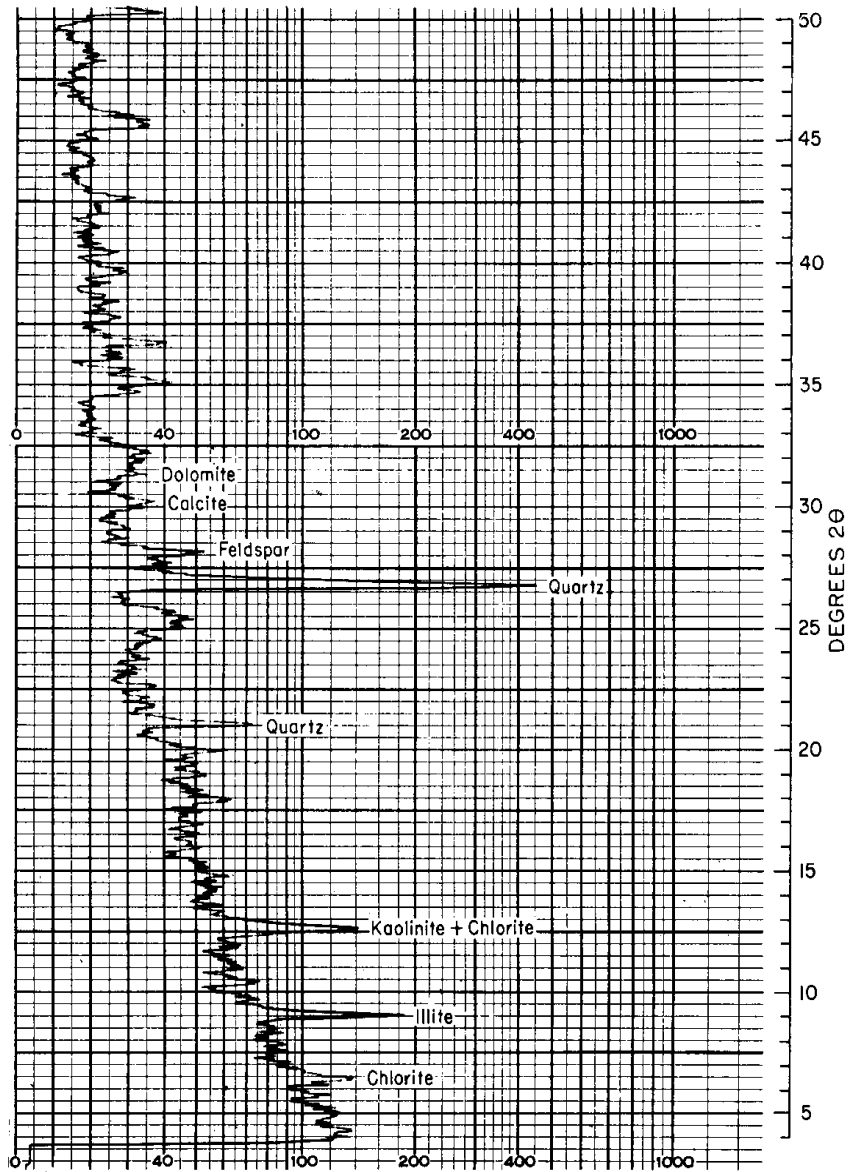


Figure 5. X-ray diffractometer trace of sample 12.

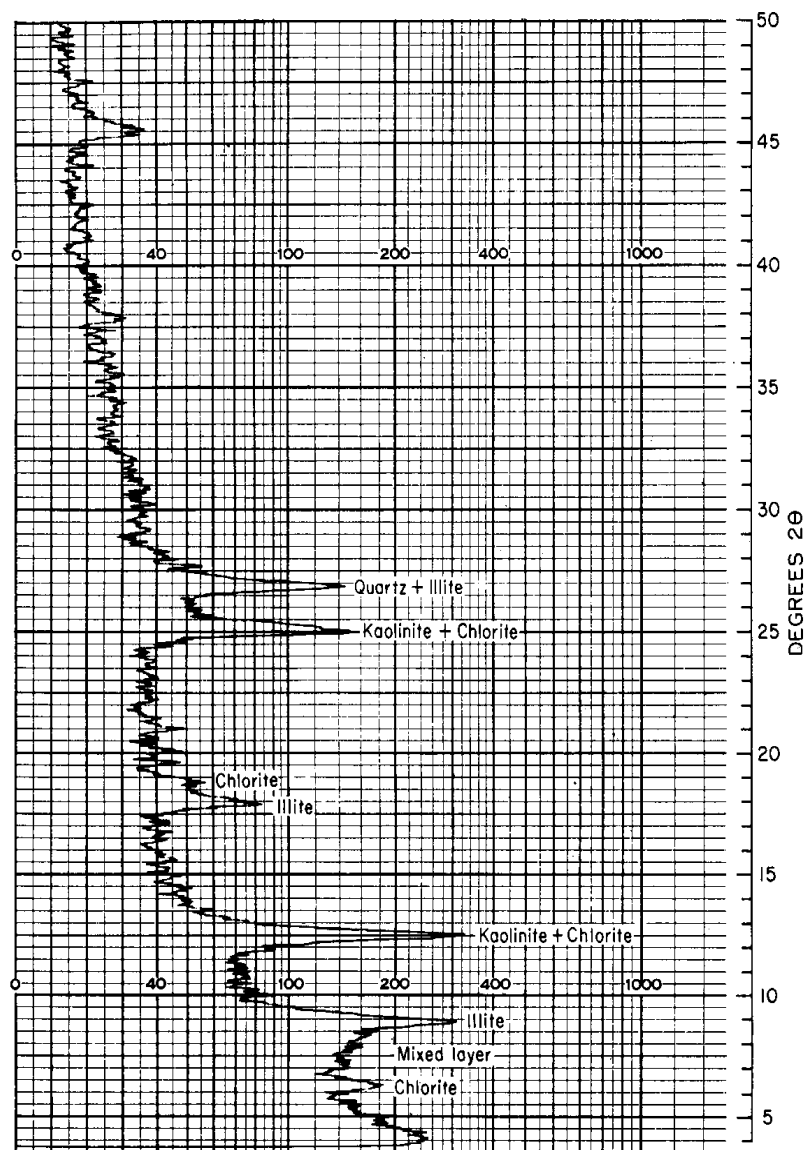


Figure 6. X-ray diffractometer trace of $<2\mu$ -fraction of sample 12.

Table 7.—Major mineral constituents of samples

Sample No.	Quartz (pct.)	Feldspar (pct.)	Calcite (pct.)	Dolomite (pct.)	Pyrite (pct.)	Illite (pct.)	Kaolinite (pct.)	Chlorite (pct.)	Mixed lays (pct.)
1----	20	5	--	10	--	40	6	6	13
2----	15	5	15	10	--	35	5	10	5
3----	15	5	5	5	--	40	5	10	15
4----	15	5	5	35	--	35	--	--	5
5----	20	5	5	5	--	35	10	10	10
6----	20	3	--	7	--	25	15	10	20
7----	40	10	--	--	--	25	10	10	5
8----	25	5	2	3	--	24	12	16	13
9----	10	5	5	8	--	30	12	18	12
10----	15	5	5	--	--	48	8	8	11
11----	15	5	--	--	--	30	15	20	15
12----	20	5	5	5	--	25	12	12	16
13----	25	5	--	5	--	25	15	10	15
14----	20	3	5	--	--	36	12	12	12
15----	25	4	3	3	--	20	15	10	20
16----	25	5	5	5	--	24	10	16	10
17----	25	5	5	--	--	45	10	5	5
18----	20	5	5	5	--	45	10	5	5
19----	20	5	5	5	--	30	10	10	15
20----	20	5	3	2	--	--	10	10	50
21----	25	--	3	2	--	--	15	--	55
22----	20	5	--	--	--	55	6	6	8
23----	20	3	--	10	2	55	5	--	5
24----	20	8	--	7	--	35	7	7	16
25----	25	5	--	--	--	36	12	12	10
26----	20	5	3	--	3	36	12	12	9
27----	20	5	5	--	--	15	15	15	25
28----	20	5	3	3	3	50	2	--	14
29----	20	5	2	3	--	52	4	4	10

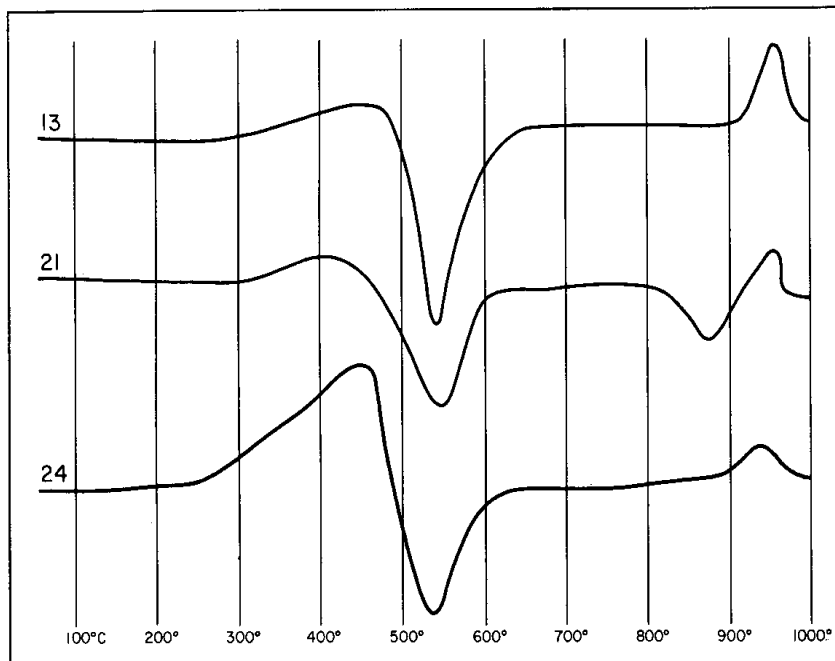


Figure 7. Differential thermal analysis curves of three representative samples (13, 21, and 24).

high sulfur content. Sample 20 had a high percentage of infixed layer illite and chlorite, some fine-grained calcite, a moderate amount of organic material, and a high sulfur content. A high percentage of illite, some pyrite, abundant organic material, and a high percentage of sulfur were found in sample 28. Sample 3 had a high percentage of illite, some fine-grained calcite, abundant organic material, and a high sulfur content.

As shown in table 6, feldspar was present in most of the samples. In all probability it acts as a flux in the vitrification process, and it vitrifies in the range of temperatures used in the bloating process. A mineral that will vitrify rapidly is as necessary as a material that will produce volatiles and cause bloating.

FAVORABLE AREAS FOR EXPLORATION

Analyses of shale samples have indicated that some areas in Indiana should be explored further as possible sites for lightweight aggregate plants. Other factors, such as thickness of shale, depth

of overburden, location of markets, and availability of rail facilities and fuel, also must be evaluated. Favorable areas for exploration of lightweight aggregate potentialities are shown on plate 1 by Roman numerals I through IX. These numerals do not indicate which areas are most favorable but serve only to identify the areas. In general, the most favorable areas are south of the Wisconsin glacial boundary, but some areas north of this boundary are accessible because of a thin cover of glacial drift. Wayne (1956) has outlined the thickness of drift in Indiana north of the Wisconsin glacial boundary.

Area I is in southeastern Newton County, southern Jasper County, and northwestern White County. The New Albany shale crops out in this area and has a rather thin cover of glacial drift (Wayne, 1956). A brick plant at Wolcott in White County, abandoned in the 1930's, reportedly operated a pit that contained 40 feet of New Albany shale. The authors have seen 12 to 15 feet of New Albany cropping out in stream banks just north of the town of Remington in southern Jasper County. As previously pointed out, the test results (table 2) for sample 3 were excellent. Rail transportation is available in this area, and the market in northern Indiana and Illinois should be able to absorb large tonnages of lightweight aggregate.

Area II is in Montgomery, Tippecanoe, Fountain, and Warren Counties. Undifferentiated shales of the Borden group crop out in this area of rather thin drift (Wayne, 1956). Brick plants at Crawfordsville and Attica use shales of the Borden group. Samples 5 and 7 from these two locations bloated. Rail transportation is accessible in this area, and further testing of samples may show that Area II would be a desirable location for a lightweight aggregate plant.

Area III is in southern Vermillion County and northern Vigo County. Two shale samples from this area bloated; one was from the shale above Coal IIIa, and the other from the shale above Coal VII. Many strip mines and exposures are found in this area of thin glacial drift (Wayne, 1956). Rail transportation is available on the periphery of Area III.

Area IV is in Morgan County and southern Hendricks County. The Wisconsin glacial boundary crosses this area. Outcrops of shales of the lower Borden group are numerous south of Mooresville, and a few outcrops have been found in stream banks in Hendricks County. Midwest Aggregates, Inc. is now producing lightweight aggregate from the New Providence shale of the Borden group in this area.

Area V is in Knox, Daviess, Pike, Gibson, and Warrick Counties. Although samples from this area did not give as good test results as some others, the abundant shales of the Allegheny and Conemaugh series warrant further exploration. The rather thick shales

which are found above the Lower and Upper Millersburg coals in this area show possible use as raw material for lightweight aggregate. Rail transportation is accessible in many parts of the area.

Area VI is in Lawrence County and northwestern Orange County. Samples 20 and 21, collected from shales in the Bethel formation in this area, gave excellent test results. The shale is rather thin (table 1), but thicker shales may be found after further exploration. Two railroads cross the area.

Area VII is in Jackson County and northern Washington County. Two brick plants, one at Brownstown and the other at Medora, operate pits in the New Providence shale of the Borden group, which crops out in this area. No samples were collected from the New Providence shale in this area because samples of New Providence shale obtained both north and south of this area gave satisfactory test results. The New Providence shale, a persistent lithologic unit, in all probability would give good test results if sampled in this area. Two railroads cross Area VII.

Area VIII is in parts of Bartholomew, Jackson, Jennings, Jefferson, and Scott Counties. The New Albany shale is exposed at many places in this area. Sample 23, collected near North Vernon in Jennings County, gave excellent test results. Inasmuch as the New Albany shale is rather thick and does not vary greatly in its lithologic characteristics, other samples of it probably would give essentially the same results. Rail transportation is available in this area.

Area IX is in Clark and Floyd Counties and covers exposures of both the New Albany and the New Providence shales. Both units were sampled (samples 28 and 29) and both bloated. Rail transportation is available in this area.

As shown above, some areas in Indiana can be considered favorable locations for lightweight aggregate plants using shale as raw material. Each area would need to be evaluated as to geologic formations, potential market, and available transportation. Pilot plant testing would have to be made in order to evaluate fully the bloating characteristics of the shales.

CONCLUSIONS

1. Many shales in Indiana are potential sources of manufactured lightweight aggregate; 21 of the 29 tested samples bloated. The New Albany, New Providence, and Bethel shales and the shale above Coal VII in the Dugger formation bloated best.

2. The range in chemical composition in which bloating occurs, as determined by Riley (1951), is valid. Some shales within this

range do not bloat, however, because other factors, such as particle size and mineral composition, are important to bloating.

3. Ternary composition diagrams based on the major constituents do not separate shales that bloat from those that do not; this substantiates the conclusion that the cause of bloating is complex and cannot be attributed to any single factor.

4. Such volatile substances as CO₂, SO₂, H₂O, O₂, and perhaps hydrocarbons can cause bloating.

5. Particle size of minerals is a major factor in bloating. Large discrete particles of pyrite, dolomite, and calcite result in nonuniform bloating, but fine-grained, evenly dispersed particles of these same minerals produce uniform bloating.

6. X-ray diffraction and differential thermal analyses must be used to supplement each other in determining mineral composition of shales.

7. The minerals calcite, dolomite, pyrite, and goethite and the clay minerals illite, chlorite, and kaolinite can release volatiles and thus contribute to bloating. In addition, the oxidation of organic material can produce volatile substances.

8. Such minerals as calcite, dolomite, and pyrite must be present in shales only in limited amounts. Too much gas prevents bloating because the pressure is too great and thus the gas breaks through the viscous glassy layer around the shale particle. A mineral like feldspar, which vitrifies rapidly at temperatures between 2,100° and 2,300° F., must be present to form a viscous glassy jacket around the particle to hold the expanding volatiles and cause vesiculation.

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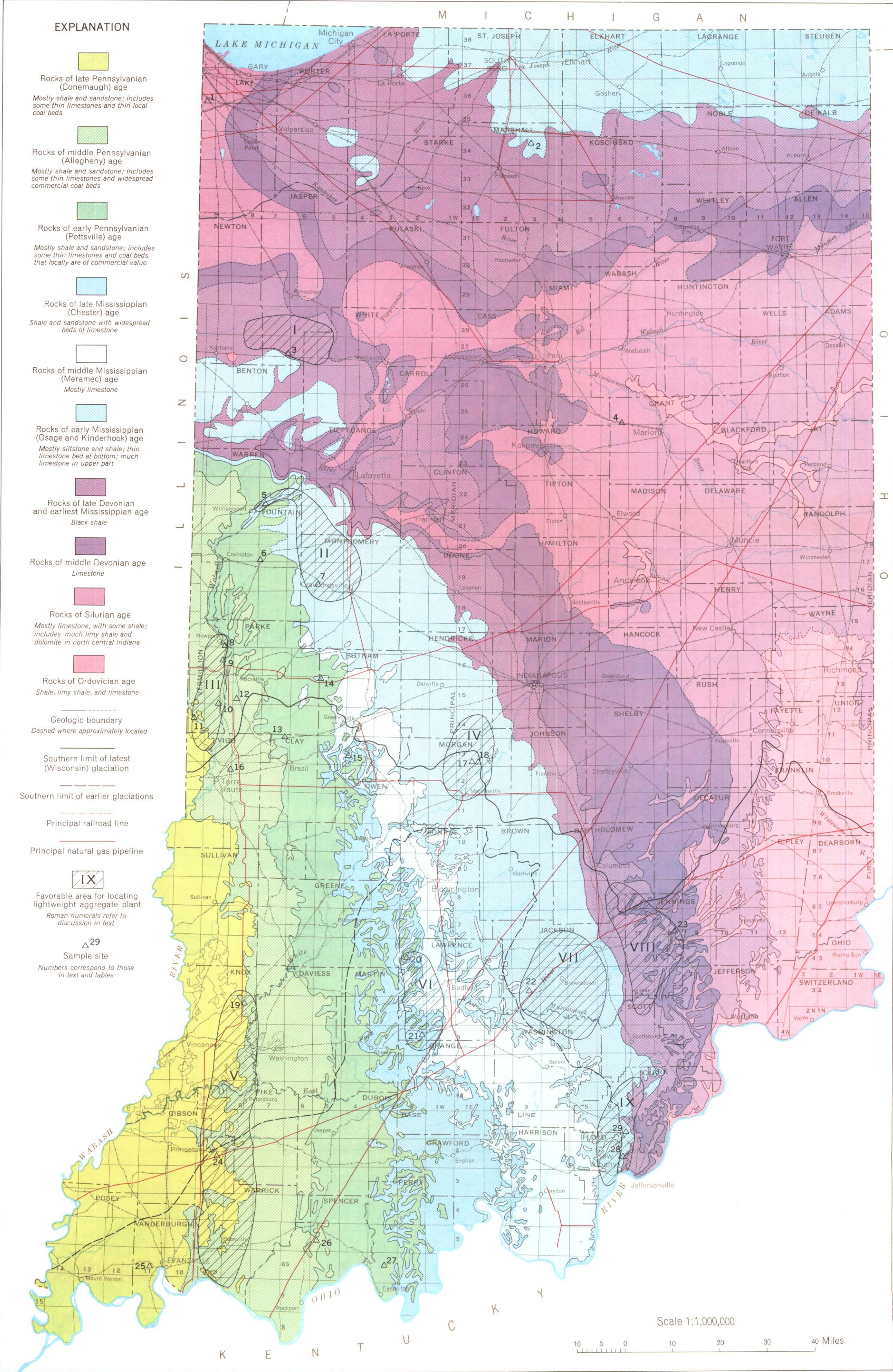
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Base modified from U. S. Geological Survey Base map of Indiana, 1953.

MAP OF INDIANA SHOWING LOCATION OF SAMPLE SITES, GENERALIZED GEOLOGIC BOUNDARIES, AND FAVORABLE AREAS FOR LIGHTWEIGHT AGGREGATE PLANTS

Geologic boundaries modified from Indiana Geological Survey Atlas Map 9, 1956.