Short Papers on Research in 1967

Edited by Doris E. Zeller

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OF
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BULLETIN 191, PART 1

1889
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of KANSAS

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Short Papers on Research in 1967

This collection of papers continues the series begun in 1967 as a part of the *Reports of Studies* in the Bulletin. The papers present scientific and economic results of work by members of the State Geological Survey of Kansas and others during the calendar year 1967.

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O. KARMIE GALLE
ERNEST E. ANGINO

Sr

Trace Elemental Analysis of Fresh Water by Atomic Absorption

ABSTRACT

In studies of water quality, knowledge of trace element composition is important. Cobalt, iron, lithium, manganese, nickel, strontium, zinc, and silicon were determined in water from several streams and ponds of the Kansas River Basin. The method has proven to be routinely useable in the range of 0.01-1 ppm for Co, Ni, and Li, 0.01-10 ppm for Mn, Sr, Zn, and Si, and 0.01-100 ppm for Ca, Mg, and Fe. These trace elements and others can be determined from the same concentrated solution by evaporating 500 ml of filtered water down to 10 ml. This solution is then brought up to 50 ml by the addition of distilled, deionized water. The coefficient of variation for each element was: Co 6% at 0.08 ppm, Fe 5% at 0.06 ppm, Li 10% at 0.01, Mn 2.5% at 0.02, and Sr 7% at 0.08 ppm levels, respectively. While it is recognized that these coefficients of variation values can be improved, they were deemed adequate for this study.

A study of the trace element content of the rivers in the lower Kansas River Basin was undertaken as part of a much larger program directed toward developing a programed water quality model (O'Brien, *et al.*, 1967) for the main stem Kansas River.[1] The purpose in doing the trace element analyses was two-fold: (1) To develop methods for trace elemental analyses, and (2) To collect data on the trace element content of the different rivers in the Kansas River Basin. Because little information on trace elements exists for the rivers of this area, it was thought that such an investigation would be worthwhile.

The rivers of the basin which were sampled for this study (Fig. 1) include: the Smoky Hill, Saline, and Solomon rivers in the western part of the area, the Republican and Blue rivers in the central part, and the Delaware and Wakarusa rivers in the eastern part. The Kansas River, which extends from Junction City to Kansas City, was also sampled at intervals of 20 to 40 miles.

All samples analyzed for trace elements were prepared in the following manner. Upon being received in the laboratory the water was filtered through a 1.2 μ Millipore filter to remove any suspended material. Five hundred ml of the filtered water (to which was added 10 ml of hydrochloric acid) was then evaporated down to a volume of approximately 10 ml, transferred to a 50 ml volumetric flask, and diluted to the mark with distilled, deionized water. This gave a solution with a concentration factor of 10. The acid was added to keep the SiO_2 and iron in solution. Diluting the concentrated water to 50 ml with methanol was also tried. According to Wheat (1964), the use of an 80 percent water-methanol solution will increase the sensitivity three to four times over straight aqueous solutions. This can be helpful where low concentrations of a particular element are present. However, a drawback exists to the use of methanol solutions. It was found that when methanol was used as a diluent, fresh standards had to be made up for each series of analyses. With aqueous solutions, standards, which ranged from 0.1 to 10 ppm, could be kept an average of 5-8 weeks before any change was noted. Iron was an exception to this average. Solutions of 100 ppm or less could not be kept for more than one day. For the bulk of the samples run during this study, the methanol dilution step was not necessary. Adequate sensitivity was obtained using the aqueous solutions. The elements routinely determined on the concentrated water solutions were Co, Fe, Li, Mn, Ni, Si, (as SiO_2), Sr, and Zn. In addition to these elements, Ca and Mg

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[1] Financial assistance for part of this work was provided by a Federal Water Pollution Control Administration grant to one of us (W. J. O'Brien with E. E. Angino) under contract PH 86-66-63 to the Center for Research in Engineering Science, The University of Kansas. T. C. Waugh aided in the development of the method for trace element analysis, and R. L. Kennedy, R. L. Mudd and Mrs. Khawla Sabih prepared the samples.

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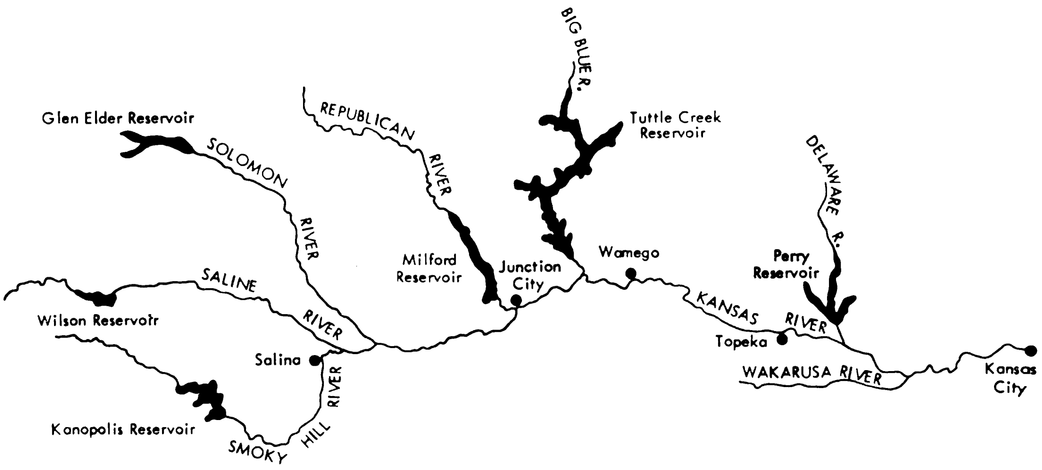

FIGURE 1.—Map of the portion of the Kansas River Basin from which samples were collected.

were determined separately. Due to the high concentration of Ca and Mg, it was necessary to dilute the original water sample 25 times in order to complete a successful instrumental analysis.

The instrument used for the determinations was a Jarrell-Ash model 82-500 series atomic absorption spectrometer. The instrument utilizes a 0.5-m Ebert monochromater and, for our determinations, was equipped with a 100-μ entrance and a 150-μ exit slit. The burner used was a Jarrell-Ash triflame burner which consists of a laminar flow head mounted over a Hetco burner. A fuel combination of air-H_2 was used for all determinations except SiO_2. A fuel combination of acetylene-nitrous oxide was necessary to determine SiO_2 successfully. Fuel and support gas pressures were adjusted for maximum sensitivity for each element.

Twelve locations within the Kansas River Basin were sampled once every month from June 1966 through May 1967. The elements studied, the concentration ranges measured, and the calculated coefficient of variation for each element are summarized briefly in Table I.

The primary interference encountered in the determination of calcium and magnesium came from sulfate (SO_4). The sulfate content of the river waters was as high as several hundred ppm at some locations. This interference was eliminated with the addition of 1500 ppm strontium as $SrCl_2$. There were no interferences encountered during this study which were not eliminated by the addition of strontium.

One of the interferences which was found to be of importance was the effect of different acids upon the sensitivity of calcium and magnesium in a hydrogen-air flame. Galle and Angino (1967) reported a difference in standard curves obtained when four different acids were used to put the calcium and magnesium into solution. The acids studied were $HClO_4$, HCl, HNO_3, and H_2SO_4. The work was done with a Hetco (total consumption) burner and a hydrogen-air fuel mixture. Figures 2 and 3 show the results obtained for calcium and magnesium, respectively. It was felt that this effect was probably caused, in part, by the type of burner in the system. It was also felt that the use of a laminar flow type burner would eliminate much of the differences demonstrated by these two figures. However, this is not true if a low-temperature hydrogen-air flame is used. Figure 4 shows the results obtained using the same concentrations of calcium as shown in Figure 2 but doing the determinations on a laminar flow type burner and using a hydrogen flame. As can be seen, the differences still remain, but the net effect was to increase the sensitivity in each case. As is com-

TABLE 1.—Concentration ranges and calculated coefficients of variation of the elements studied.

Element	Concentration range, ppm	Coefficient[*] of variation
Ca	10.0 - 176.0	....
Mg	8.0 - 54.0	....
Sr	0.012 - 0.3	7.0% at 0.08 ppm level
Co	0.0 - 0.3	6.0% at 0.08 ppm level
Ni	0.003 - 0.12	....
Fe	0.013 - 0.8	5.0% at 0.06 ppm level
Mn	0.003 - 0.7	2.5% at 0.02 ppm level
Zn	0.01 - 0.5	0.35% at 0.40 ppm level
Li	0.0 - 0.04	10.0% at 0.01 ppm level
SiO_2	5.9 - 31.3	2.3% at 7.0 ppm level

[*] Coefficient of variation was calculated on the basis of 12 determinations.

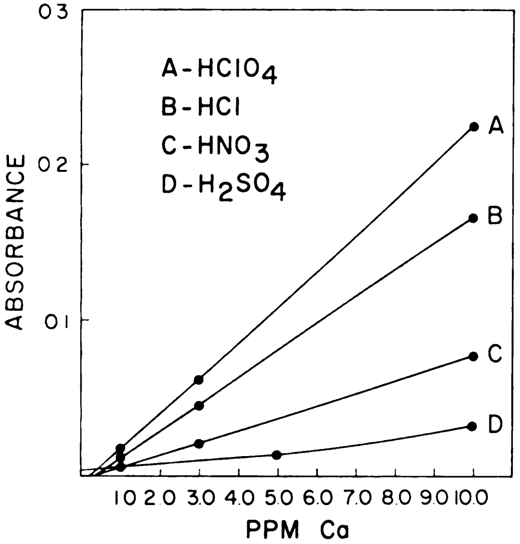

FIGURE 2.—Comparison of the effect of four acids upon the absorption of calcium using a hydrogen-air flame and a Hetco burner.

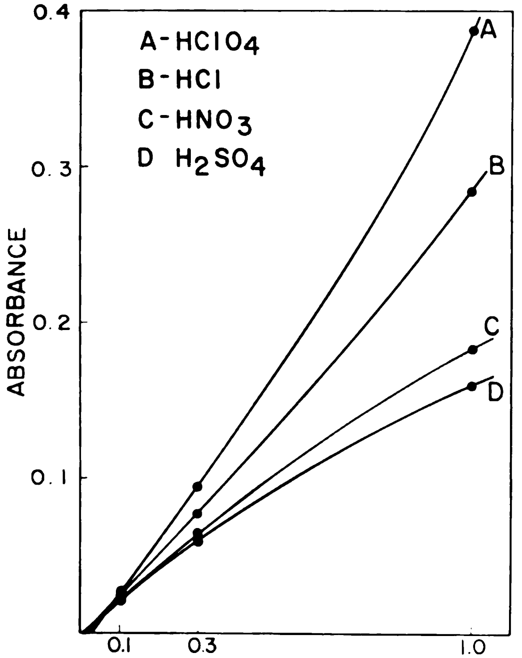

FIGURE 3.—Comparison of the effect of four acids upon the absorption of magnesium using a hydrogen-air flame and a Hetco burner.

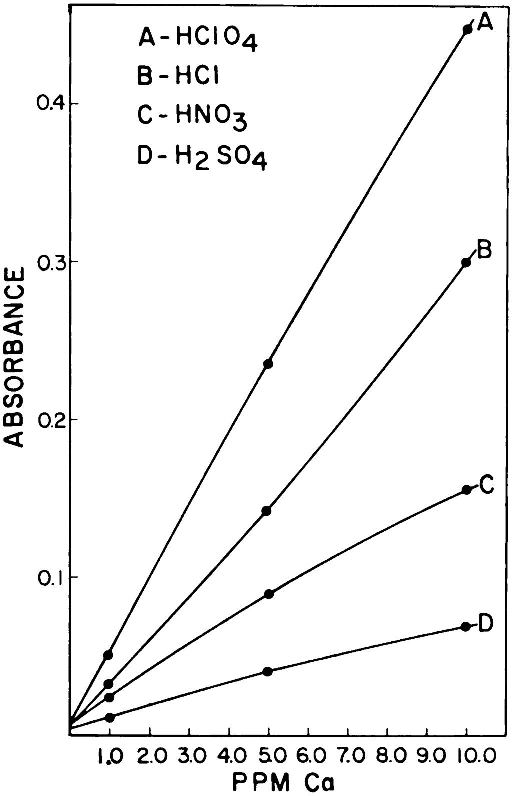

FIGURE 4.—Comparison of the effect of four acids upon the absorption of calcium using a hydrogen-air flame and a laminar flow burner.

monly known, the differences can be eliminated by use of a hotter flame (e.g., an acetylene-air flame). This eliminates the difference between $HClO_4$, HCl, and HNO_3. However, a difference in the H_2SO_4 curve remains, due, most likely, to the sulfate interference. The hydrogen-air flame was used when this work was begun and its use continued because it produces less recorder noise than an acetylene-air flame. The sensitivities obtained for calcium and magnesium are adequate for our needs and as long as standard and sample solutions are made up with the same acid, no viscosity or other problems were encountered with the Ca and Mg determinations.

The concentrations of the trace elements, as determined for this study, appear to have seasonal variations. The highest concentrations of Co, Fe, and Ni were found during the months of July, August, and September. The concentrations of these elements, in general, decreased as the weather got colder. The concentrations of Li, Mn, and Sr, however, showed a tendency to increase during the winter months of December, January, and February. The data for Zn and SiO_2 are incomplete and go back only to December of 1966; therefore, no information about seasonal variations is available for them.

A technique used for SiO_2 determinations might be of interest. The cathode power supply of the Jarrell-Ash atomic absorption units may be operated in two different modes. In the normal mode of operation a dc current drives the cathode tube which is used in conjunction with a mechanical chopper to provide a signal to the monochromater of 87 cycles per second. The second mode of operation involves changing the input current to the cathode tube from dc to pulsed dc. This is done on the Jarrell-Ash instrument by simply moving a switch from the "normal" position to what is called a "high intensity" position. In this position the signal is pulsed electronically at 87 cps. This in effect provides a signal of high intensity for short intervals maintaining a mean current below that of the normal hollow cathode rate. High intensity provides an output 5-20 times as intense as the mechanically pulsed dc output. It was found that by using the high intensity mode of operation for the SiO_2 determinations most of the signal noise in the recorder which was present when using a scale expansion of 8-10 mv was eliminated. When using the high intensity mode, the sensitivity is reduced; but when most of the determinations involve SiO_2 in concentrations of 10 ppm or greater, the reduction in sensitivity does not appear to be critical. This method was tried in all determinations in which the recorder scale expansion was needed and an excessive amount of signal noise was present (particularly in determinations of Co and Sr). The technique was successful in reducing the noise level so that readings could be made more accurately.

Another statement on SiO_2 determinations by atomic absorption can be made here. Presently, we are working on a method for the determination of SiO_2 and Al_2O_3 in carbonate and silicate rocks. This method involves fusing a powdered sample with lithium metaborate as described by Ingamells (1966). After the sample is fused, it is taken into solution with H_2O and nitric acid. The solution technique appears to be successful and our preliminary results on USBS and USGS standard samples is encouraging. The method, however, breaks down with those samples which have 80-100 percent SiO_2. In these cases it is difficult to get all of the SiO_2 into solution. However the technique appears to work for most other samples, particularly carbonates.

As far as interferences are concerned, little information in the literature exists as a guide. Calcium appears to interfere slightly and lithium in large amounts causes some interference. Figures 5 and 6 demonstrate these interferences. Iron apparently does not interefere, at least at the levels encountered during this investigation. Aluminum may be expected to cause some interference, although this has not been checked to date.

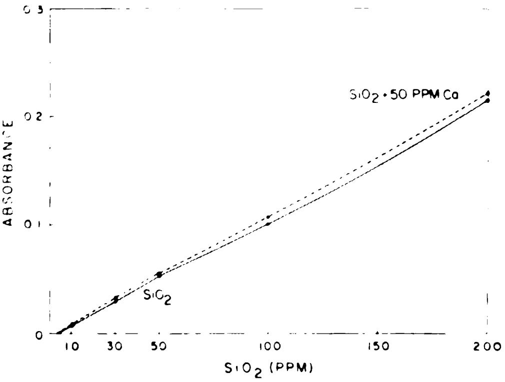

FIGURE 5.—Comparison of standard curves of SiO_2 and SiO_2 plus 50 ppm Ca.

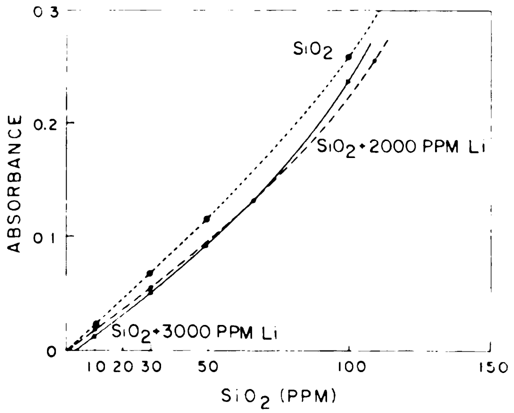

FIGURE 6.—Comparison of standard curves of SiO_2 and SiO_2 plus 3000 ppm and 2000 ppm Li.

CONCLUSIONS

The highest concentration of trace elements is found in the western part of the region, in the Smoky Hill, Saline, and Solomon rivers. The concentration of the trace elements determined, in general, gradually decreases and is the lowest at the eastern end of the region or the lower portion of the Kansas River near Kansas City. Apparently most of the trace elements found in the Kansas River itself are contributed by the Dakota Formation through which the Smoky Hill, Saline, and Solomon rivers flow.

Very little in the way of trace elements is contributed east of the point where the rivers join to form the Kansas River. The fact that the elemental concentrations are diluted by the time they reach the eastern end of the Kansas River could explain why such low concentrations of the elements are found at the eastern end of the region.

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RONALD G. HARDY
JOHN M. HUH
MAYNARD P. BAULEKE

Kansas Raw Materials for the Manufacture of Reflective Glass Beads

ABSTRACT

Cheyenne Sandstone from Comanche County, dune sand from near Liberal, Kansas, and caliche or chalk from the Fort Hays Limestone Member of the Niobrara Chalk were used in glass compositions to make reflective glass beads. Both sources of sand were acid-leached to reduce their iron content. Index of refraction and light reflectivity of the experimental glass beads produced were of the same value as those of currently manufactured commercial products.

Several years ago Nixon, *et al.* (1950) recommended that the Cheyenne Sandstone was suitable for industrial use. Nothing has occurred in the passing years to develop the deposit, mainly because of the distance from markets and competing sands from other sources in surrounding states. This is a reexamination of the Cheyenne Sandstone for possible use in the manufacturing of reflective glass beads, a growing market, which can utilize a glass of higher iron content than many of the current industrial glasses. In addition a sample of dune sand from near Liberal, Kansas, was beneficiated and compared with the Cheyenne Sandstone as a potential raw material in the manufacture of glass beads.

The Cheyenne Sandstone in Comanche County is poorly indurated. Thus, no special digging equipment is required to recover it. Hydraulic mining would be possible and could also serve as the first step in removing impurities. Dune sands are easily dug from the ground.

The laboratory beneficiation method for Cheyenne Sandstone was as follows:

1. Raw sand sample crushed by jaw crusher. Dried at 115° C. 4000 gm sample weighed.
2. Washed and scrubbed for 2 hrs: 4000 gm sand, 5000 ml tap water, 8 gm sodium hexametaphosphate.
3. Screened through 150-mesh sieve: +150 for further beneficiation; —150 discarded.
4. Acid washing of 150-mesh sand: 3400 gm sand, 1200 ml 37% technical grade HCl, 2800 ml tap water. Washed for 4 hrs; rinsed away acid; dried at 110° C.
5. Magnetic separation to remove magnetic iron.
6. Sand ready for use.

The laboratory beneficiation of dune sand was as follows:

1. Screen raw sand through 28-mesh sieve to remove rubbish.
2. Pass through magnetic separator to remove hematite and iron-cemented quartz particles.
3. Acid leaching of iron compounds: 2300 gm sand, 4600 ml 37% technical grade HCl, 4600 ml tap water. Agitate for 6 hrs in a ball mill without balls.
4. Wash and neutralize the acid.
5. Sand ready for use.

In addition to the Kansas sands, local calcium carbonate sources (Fort Hayes limestone and caliche) were used in glass compositions. The limestone and caliche were used without any beneficiation.

Numerous glass compositions were melted, but only a few representative ones are listed in Table 1. Some of the compositions may be uneconomical for the manufacture of glass beads,

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TABLE 1.—Typical glass compositions made with Kansas sands and calcium carbonates.

Sample no.	Composition, in percent										
	Beneficiated Cheyenne Sandstone	Raw dune sand	Fort Hays Limestone Member	Caliche	Li_2CO_3	Anhydrous boric acid	Na_2CO_3	K_2CO_3	As_2O_3	$BaCO_3$	ZnO
KGS-2A	61.3	......	12.9	......	4.3	0.4	4.7	15.7	0.8	......	......
KGS-3B	42.0	......	0.3	......	3.8	......	......	12.0	0.1	17.6	3.7
KGSPE-2	......	35.8	9.3	35.0	......	......	19.9	......	......	......	......

but they were used mainly to check our laboratory procedures and equipment for making glass beads. Part of the melted glass composition was poured into a graphite mold to form a sample of glass and part was poured into water to form a crushable frit for later processing into beads.

If a free-falling particle of glass is heated to its "melting" temperature, the surface tension of the viscous glass will shape the particle into a perfect sphere. All test glass beads were formed by dropping the glass particles into a verticle furnace (Fig. 1) using the flame fusion method.

The test glass frits were ground in a porcelain ball mill using alumina balls and screened to —32 +65 mesh. It is important not to have too much variation in particle size. If the particle is too large it will fall through the flame, if it is too small the velocity of the hot gases will prevent the particle from reaching the hot flame. For proper fusion, the particle must be in the flame long enough to soften the glass to a point where the surface tension forces can shape the sphere and produce a smooth surface. Too low a temperature will produce only partial spheroidization and a "wrinkled" surface. Each glass composition and particle size range has its best spheroidizing temperature and gas velocity.

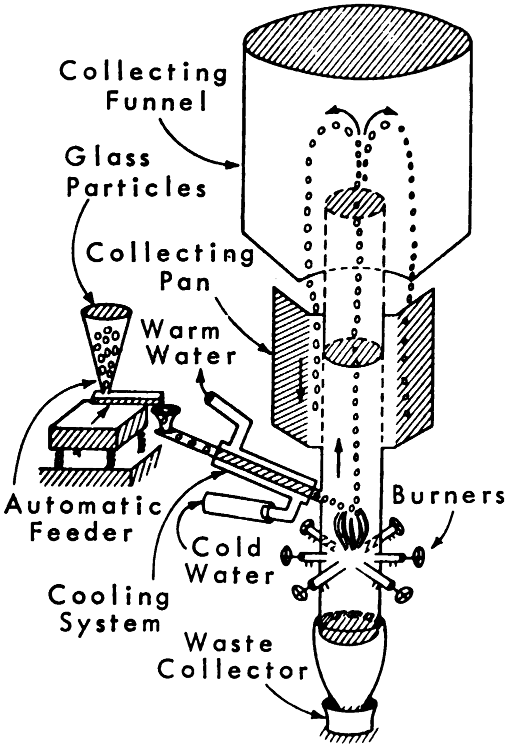

FIGURE 1.—Sketch of apparatus used in making glass beads.

After grinding and screening, the glass particles were fed into the furnace about 10 inches above the burner. The feeder tube is water-cooled to prevent the glass particles from sticking at the furnace entrance. The glass particles fuse almost instantaneously in the flames and are carried upward by the velocity of the hot gases, out of the furnace tube, and fall into the collecting funnel, and roll onto a sloping collecting plate that collects the beads and also somewhat classifies them as rounded or non-rounded. A vibrating table can be used for further separation of imperfect beads.

Index of refraction and percent daylight reflection values are listed in Table 2. For ordinary highway useage, beads must have an index of refraction greater than 1.50 and a percentage of daylight reflection greater than 45. Several compositions were made with unbeneficiated dune sand. The higher iron content produced more color in the glass and reduced the percent daylight reflectance value.

TABLE 2.—Physical properties of glass produced from Kansas sands and calcium carbonate.

Sample no.	Color	Index of refraction*	Percent daylight reflection†
KGS-2A	clear	1.51	63
KGS-3B	light blue tint	1.58	50
KGSPE-2	light green tint	1.57	45.7

* Measured by the comparative oil immersion method.
† Test done by Materials Testing Laboratory, Kansas State Highway Commission. MgO is rated as 100% daylight reflection.

Crushing the frit into proper particle size is a major problem, as the percentage yield of correct particle size is small. An interesting future study would be the quenching temperature variables on the fracture characteristics of glass. There are some indications, from the manner in which thick sheets of tempered glass break, that a "heat treating" technique could be developed that would permit the glass to fracture into particles of uniform size, thus producing glass beads of a uniform size.

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MAYNARD P. BAULEKE
CLARENCE S. EDMONDS

Thermal Expansion of Siliceous Kansas Clays (Dakota Formation)

ABSTRACT

Thermal expansion data are presented for eight siliceous clays from the Dakota Formation in Kansas. In clays that contain high percentages of free quartz, the negative thermal expansion behavior of the β-quartz exerts a controlling influence on the thermal expansion of the fired clay.

Difficulties were found in glazing test bricks made from siliceous clays from the Dakota Formation in a one-fire glazing operation requiring a temperature of 2300°F (cone 9). The matured glaze would pop off curved edges and even come loose from flat surfaces. Only a large mismatch in the thermal expansion of the glaze and the fired clay could cause such a condition. It was decided to measure the thermal expansion of eight selected clays from the Dakota Formation (Table 1).

X-ray diffraction methods identified the major clay mineral as kaolinite and the major impurity as quartz (SiO_2) in each clay sample.

Thermal expansion is defined as the $\Delta L/L$ per degree temperature change. ΔL is the change in length per degree of temperature change. L is the original length. Units of thermal expansion are in/in/°F or cm/cm/°C or percent length change. Percent length change is preferred as it does not require a conversion factor in order to compare data measured by two different temperature scales. All data in this study are reported as percent change of length.

Eight clays from the Dakota Formation of Central Kansas were extruded into one-half-inch rods, dried, and fired to appropriate temperatures. Numerous two-inch test specimens were cut. Expansion data were taken using a Gaertner Scientific Corporation quartz-tube dilatometer model D-1200. A specimen was placed in a fused quartz tube surrounded by a heater and heated at 5°C per minute. The expansion of the specimen was transferred outside the hot zone by a fused quartz rod and was measured by a dial gauge to the nearest 0.0001 inch. No correction was made for the slight expansion of the quartz rod.

A thermal expansion measurement was considered satisfactory if the dial gauge returned to the original zero point setting after the measured specimen had cooled to room temperature. If the dial did not return to zero the specimen was remounted and the test repeated.

TABLE 1.—Chemical analyses of clays from the Dakota Formation.

Code	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	L.O.I.
C-52	70.15	19.35	1.46	1.22	0.22	0.06	0.75	0.09	6.57
C-53	69.05	20.09	1.67	1.00	0.30	0.16	0.92	0.07	6.68
C-77-B	77.55	14.99	1.59	1.48	0.18	0.14	0.61	0.04	5.30
C-77-C	72.42	15.93	3.56	1.30	0.20	0.16	0.80	0.06	5.44
El-60-6	73.26	17.86	0.65	1.25	0.14	0.15	0.06	0.05	6.48
O-5-6	62.98	23.15	3.05	1.35	0.30	0.16	0.92	0.11	7.94
O-40-3	76.43	14.37	1.38	1.57	0.26	0.22	0.58	0.14	4.83
El-TC-2-10	76.71	13.82	1.43	1.27	0.30	0.29	0.93	0.09	4.84
theoretical kaolinite	45.5	39.6							13.9

Analyses were done by the Geochemistry Section of the State Geological Survey of Kansas.
C—Cloud County; El—Ellsworth County; O—Ottawa County.

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An average thermal expansion value for a wide temperature range is convenient for structural design calculations but is of little use in designing a thermal expansion match for glazes or other coatings. The presence of any crystallographic inversions are obscured in averaged data unless care is used in selecting the averaging range. It is usually desirable to report thermal expansion data graphically for the entire curve or as average values for several temperature increments so that any non-linearity of the expansion behavior may be detected. Because the shape of the thermal expansion curve is similar for all the clays, only one sample is presented in Figure 1. A thermal expansion curve for Kentucky kaolinite is shown for comparison. In Table 2 the data are divided into two sections, one below 1157°F (625°C) and the other above. The lower temperature range reflects the effects of the quartz (α-β) inversion reaction. The higher temperature range reflects the effects of the negative thermal expansion of the β-quartz phase. Note the low values for the thermal expansion.

The thermal expansion curve clearly shows why there was trouble with a high-temperature glaze adhering to the fired siliceous clays from the Dakota Formation. The problem was caused by excessive free quartz. Quartz is an unusual mineral in that it undergoes a change in crystal structure at 575°C. A change in the size of the quartz grain accompanies the change in crystal structure, as shown in the thermal expansion curve for quartz (Fig. 2). But what is more significant is that the new β-quartz structure has a negative thermal expansion; that is, it shrinks as the temperature increases. X-ray diffraction analysis of the clay after firing showed that a large amount of free quartz was present. The combined expansion of the kaolinitic clay mineral and the shrinkage of the β-quartz mineral above 625°C produce a net result that is almost flat on the thermal expansion curve.

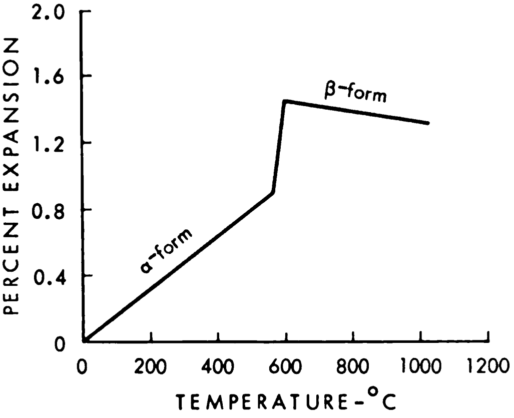

FIGURE 2.—Thermal expansion of quartz (after Searle and Grimshaw, 1959, p. 723).

It is impossible to apply a glaze to such a material on which the glaze becomes rigidly attached to the clay at temperatures above 650°C. The severe mismatch caused by the shrinking of the glaze and the non-shrinking of the clay can be severe enough to cause the glaze to come loose from the fired clay.

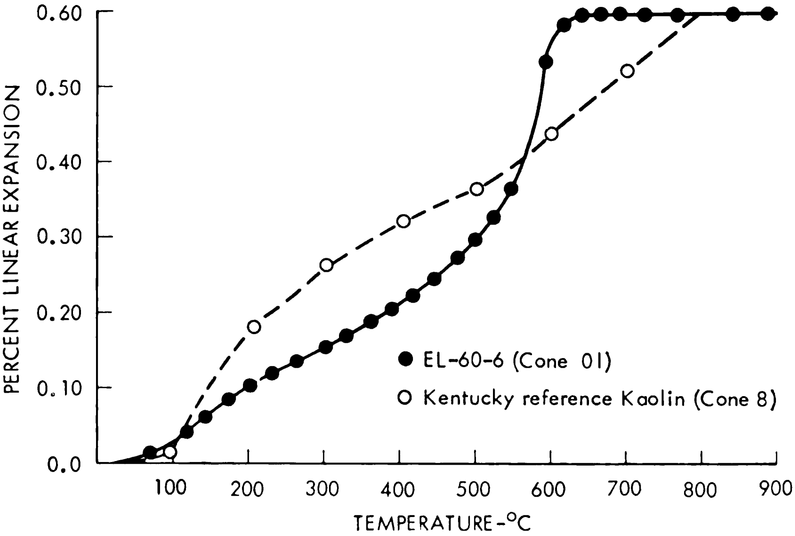

FIGURE 1.—Typical thermal expansion curve of a siliceous clay from the Dakota Formation compared with a sample of kaolin from Kentucky.

TABLE 2.—Percent linear thermal expansion of fired siliceous clays from the Dakota Formation in Kansas.

Clay code no.	1118°C (2043°F) Cone 01		1200°C (2300°F) Cone 9	
	(625°C) RT to 1157°F*	(625-950°C) 1157-1742°F	(625°C) RT to 1157°F*	(625-950°C) 1157-1742°F
C-52	0.37	0.07	0.43	0.11
C-53	0.37	0.04	0.57	0.06
C-77-B	0.45	0.04	0.56	0.07
C-77-C	0.48	0.03	0.56	0.06
El-60-6	0.59	0.01	0.56	0.02
O-5-6	0.32	0.12	0.51	0.13
O-40-3	0.53	0.05	0.53	0.13
El-TC-2-10	0.44	0.03	0.63	0.06
Kentucky kaolin			0.46	0.20

[*] RT=room temperature.

Only a low-temperature glaze, one that is still plastic or slightly fluid at temperatures above 625°C, should be used on such clays. The expansion curve below 625°C can easily be matched to that of a glaze.

SUMMARY

The thermal expansion behavior of fired siliceous clays from the Dakota Formation is influenced by the amount of free quartz present. At temperatures above the inversion of α- to β-quartz the overall rate of increase in thermal expansion is small compared with the rate of expansion below the inversion temperature. Two distinct thermal expansion regions exist. A glaze designed for matching the expansion of one region will be completely unsatisfactory for the other region.

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JOHN M. HUH
RONALD G. HARDY
NORMAN PLUMMER
MAYNARD P. BAULEKE

Testing of Kansas Clays or Shales for Non-Ceramic Utilization

ABSTRACT

Kansas clays have been extensively tested for ceramic use. A series of tests to evaluate non-ceramic uses has been instigated and indications are that certain clays or shales may have application as filler or for use as agri-chemical carriers.

Over the years a fairly extensive program of testing Kansas clays and shales for ceramic use has been carried on[1] but only a few special experiments[2] have been directed primarily toward observing non-ceramic end-use properties of these resources. Speculation on the possibility of using Kansas clays for parting agents, fillers, diluents, extenders, and as carriers for agri-chemicals resulted in our conducting a series of tests aimed toward measuring their properties for these purposes.

The first operation in the testing program is a clay-water slurry preparation, with separation of the suspended material from the sediments that drop out. Electrolytes may or may not be used, depending upon the test series or a particular clay. This treatment is particularly effective on the high-kaolin clays from the Dakota Formation of Kansas, resulting in increased alumina content and occasional lightening of color. Most of the free quartz is removed by this procedure. The flow chart for this process is shown in Figure 1.

The second operation consists of four parts performed on appropriate quantities of the dried suspension as removed during the previously described beneficiation. These four parts consist of (1) the evaluation of the material as a filler by color, rub-out, and bleach tests, (2) suspension capability in water for agri-chemical carrying, and (3) closely associated with (2), the compatibility of the material with agri-chemicals. Figure 2 illustrates the steps in these tests.

The first series of tests were on (1) a Pleistocene clay having a mixed-layer type of clay mineral (A-3-2), (2) an Upper Cretaceous Kansas shale (Blue Hill Shale) having a mixture of clay minerals, i.e., kaolinite, montmorillonite, and illite in order from greatest to least amount, and (3) a Dakota Formation clay (EL-60-6 and 13) consisting of mostly kaolinite with traces of illite and free quartz.

These tests appear satisfactory for screening clays or shales for a variety of non-ceramic uses. It is planned to examine a number of additional clays and shales by this same series of tests. Bentonites and underclays found in Kansas are being investigated.

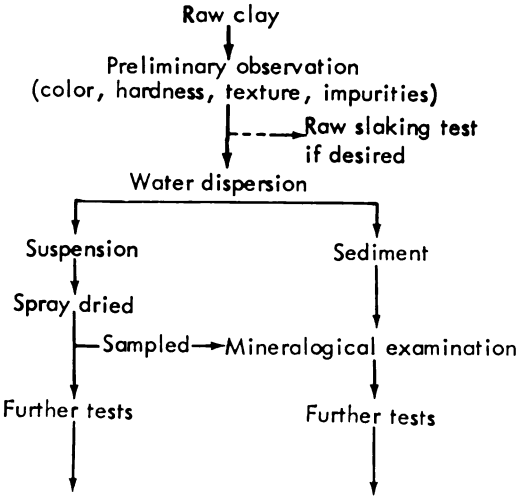

FIGURE 1.—Clay beneficiation flow sheet.

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[1] See *Bibliography*, Ceramic Use Program.
[2] See *Bibliography*, Non-Ceramic Use Program.

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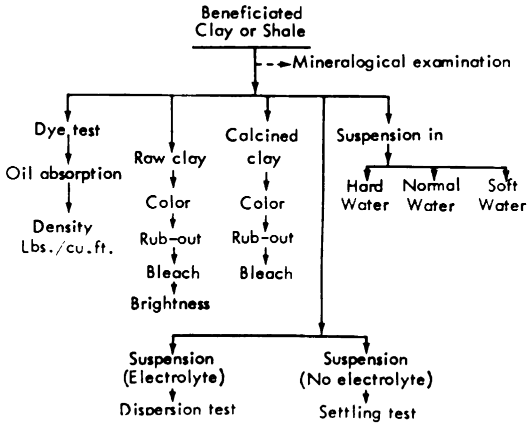

FIGURE 2.—Tests for evaluating non-ceramic properties of Kansas clays and shales.

The clays tested and described in this report give little indication of any outstanding properties for such non-ceramic uses as fillers or carriers (Tables 1, 2, 3 and 4). The high kaolin clays EL-60-6 and 13 rated the best under these tests.

TABLE 1.—Raw clay beneficiation.

Sample no. and location	Raw color	Hardness	Texture	Minerals in sediment	Clay recovery
A-3-2 Atchison County	Light greenish-yellow	Hard	Platy with yellowish stain	Mostly quartz and hematite	N.D.
Blue Hill Shale Member Osborne County	Light gray with orangish-yellow coatings	Soft	Platy	Mostly quartz some gypsum and pyrite	N.D.
El-60-6 Ellsworth County	Light gray	Hard	Smooth	Quartz and hematite	30%
El-60-13 Ellsworth County	do	do	do	do	28%

TABLE 2.—Results of tests to evaluate clays as paper, rubber, or other filler material.

Sample no.	Results of bleach test	Results of calcining on color		Rub-out test, raw material	
		750°F	1000°F	Color	Gloss
A-3-2	Very little color change	Reddish-brown with gray speckles	Dark red	Greenish-yellow	Good
Blue Hill Shale Member	No change	Brownish-buff	Dark brownish-buff	Greenish-yellow	Low
El-60-6	Changed to light gray	No change in color	Creamy white	Gray	Fair
El-60-13	do	Yellowish-gray	Very light ivory-white	Dark gray	Fair

TABLE 3.—Brightness test (reflectometer) on clay El-60-13 from the Dakota Formation.

Clay condition	Percent reflectance	
	Blue filter	Amber filter
Unheated	36.0	45.5
Heated to:		
750°F	65.0	75.0
1000°F	68.2	78.0
2134°F	72.0	83.0
2300°F	66.5	82.0

TABLE 4.—Results of tests to evaluate agri-chemical compatibility and carrying ability of clays.

Sample no.	Dye test results*	Oil absorption†		Bulk density, lbs./cu. ft. (80 to 100 mesh)	Suspension test‡		
		Free falling	Total		Hard water	Average water	Soft water
A-3-2	2.2%	15.0%	40.0%	83	1.6%	8.8%	20.8%
Blue Hill Shale Member	1.3	12.0	37.0	84	0.5	8.8	14.4
El-60-6	1.5	16.0	40.0	82	0.4	0.7	0.9
El-60-13	2.2	14.0	34.0	75	0.5	0.8	1.0

* Test to measure reactivity of clay with insecticides, maximum percent allowable = 9.0.
† Oil absorption figure represents percent kerosene absorbed.
‡ Percent solids in suspension at the end of 30 minutes.

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EDWIN D. GOEBEL
THOMAS L. THOMPSON[1]
TRUMAN C. WAUGH
LESLIE C. MUELLER

Mississippian Conodonts from the Tri-State District, Kansas, Missouri, and Oklahoma

ABSTRACT

Conodonts were recovered as acetic acid residues from carbonate rock samples from the Tri-State lead-zinc district ore bodies of Kansas, Missouri, and Oklahoma. The specimens are unaltered. The fauna belongs to the *Gnathodus texanus-Taphrognathus* Zone (late Osagian).

Minor amounts of galena and sphalerite were reported in sedimentary rock cores of Mississippian age in central and western Kansas by Lee (1933). Subsequent work by Thompson and Goebel (1963) and Goebel (1966) revealed that conodonts in association with galena and sphalerite were present as acid residues in some of Lee's cores, in many other carbonate rock cores, and in outcropping Mississippian rocks in southeastern Kansas. Conodonts, composed of calcium phosphate, are marine microfossils of unknown zoological affinities. Because of their rapid evolutionary changes in morphology and their worldwide distribution in diverse lithologies, conodonts are in popular use as tools of correlation by biostratigraphers. It was logical from the occurrences in Kansas to suspect that conodonts would be preserved in or near the ore bodies in the Tri-State District, and if so they could be used for correlation of these beds. A pilot study seemed in order, and a cooperative project with the Missouri Geological Survey[2] was undertaken.

Goebel (1966) reported on the petrographic features of carbonate rocks from western Kansas cores similar to those reported by Hagni and Saadallah (1965) in a study of six pull drifts in five mined areas in the Tri-State District. Some of the mines studied by Hagni and Saadallah were closed by June 1967 when this project commenced but effort was made to sample those sites still open. D. C. Brockie, E. H. Hare, and P. R. Dingess, of the Eagle-Picher Company, Cardin, Oklahoma, arranged entrance into Eagle-Picher mines and other open mines in the district. Also, Mr. Hare and Mr. Dingess suggested sample sites and accompanied the authors during the sampling.

PROCEDURE

At the sample sites shown on Figure 1, grab samples of about 3,000 g were obtained from mine walls and pillars except in pull drifts at sample locations 5A-P, 8A-F, and 34A-1. Samples were taken at approximately 20 foot lateral intervals along the walls of the pull drifts. It was hoped that these would approximate the sample sites of Hagni and Saadallah in the Westside mine in Kansas and the Little Greenback mine in Oklahoma.

No underground lead-zinc mines were in operation in June 1967 in southwestern Missouri. The Oronogo Circle lead and zinc deposit north of Webb City, Missouri, a part of the Tri-State District, was selected as a suitable surface section from which to sample because of accessibility and history of important production of galena and sphalerite. Winslow (in Keyes, 1894) described the geological occurrence of these minerals at Oronogo Circle. Missouri Geological Survey Notebook 1294-27 gives the following measured section (measured by Thompson, 1967) which was sampled. Bed numbers refer to sample numbers in Table 1.

Oronogo Circle; NE SE sec. 36, T 29 N, R 33 W, west side Jasper County Highway MM, Jasper County, Missouri; Webb City 7½′ Quadrangle (Missouri Geological Survey locality 1294-27).

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[1] Missouri Geological Survey, Rolla, Missouri.
[2] By permission of W. C. Hayes, State Geologist.

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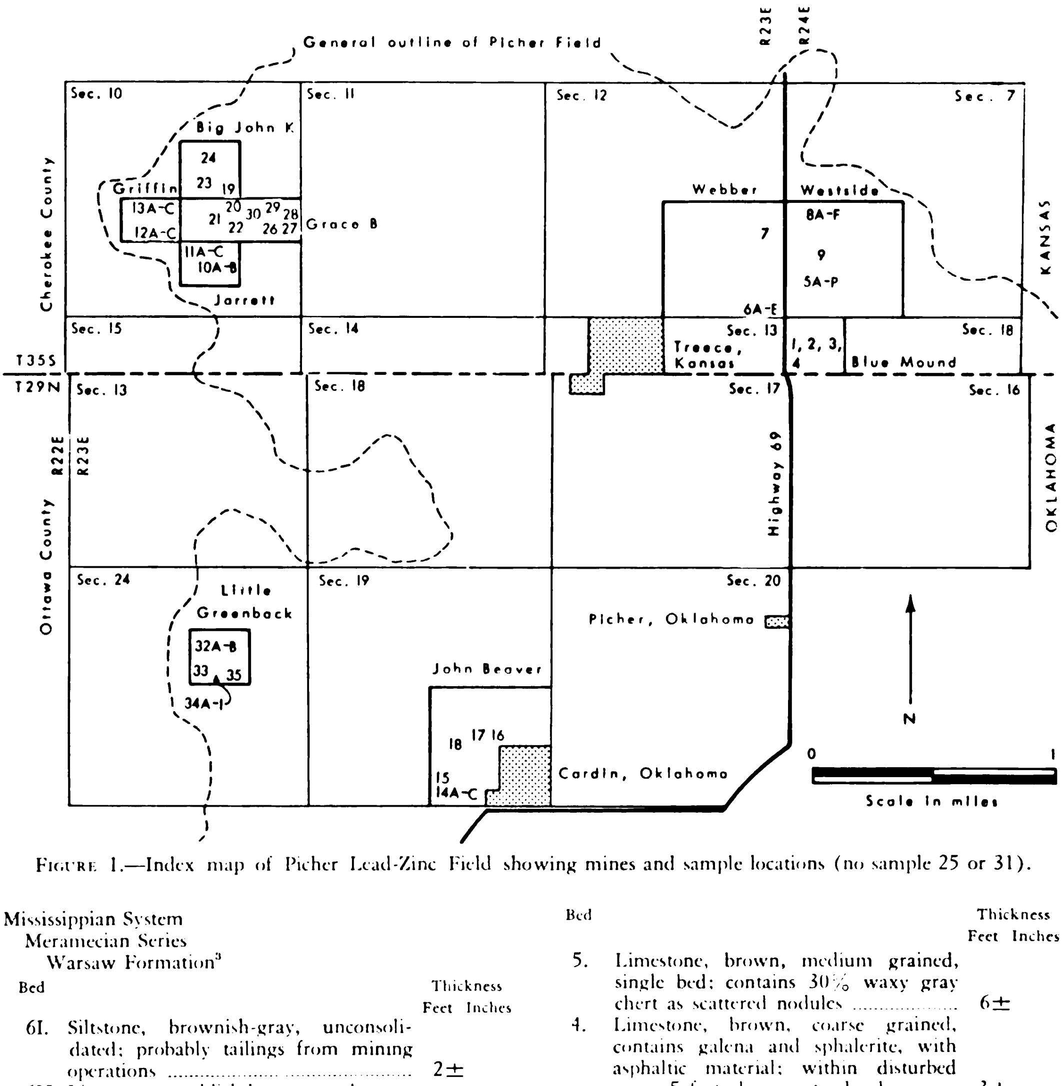

FIGURE 1.—Index map of Picher Lead-Zinc Field showing mines and sample locations (no sample 25 or 31).

Mississippian System
Meramecian Series
Warsaw Formation[3]

Bed		Thickness	
		Feet	Inches
6I.	Siltstone, brownish-gray, unconsolidated; probably tailings from mining operations	2±	
6H.	Limestone, reddish-brown to brown, weathered, tripolitic upper 5-feet	6	0
6G.	Limestone and chert; limestone light gray, medium grained; chert as intergrowths with limestone; a quasi-breccia	3	0
6F.	Limestone, very dark gray, weathers light gray, asphaltic, thick single bed	4±	
6E.	Limestone, light gray, coarse grained, very fossiliferous, good even bedding, brachiopod zones at top	2	0
6D.	Limestone, coarse grained, conglomeratic; ⅛ to ¼-inch flat pebbles included; fishtooth conglomerate lenses contain limestone pebbles	0	6
6C.	Limestone, dark gray, calcarenite, coarse grained; bedding highly disturbed	3±	
6B.	Same as unit 6C	3±	
6A.	Same as unit 6C, highly brecciated	3±	
5.	Limestone, brown, medium grained, single bed; contains 30% waxy gray chert as scattered nodules	6±	
4.	Limestone, brown, coarse grained, contains galena and sphalerite, with asphaltic material; within disturbed zone; 5 feet above water level	3±	
3.	Shale, black, platy to fissile; located laterally between units 1 and 2, not necessarily below unit 4	?	
2.	Limestone, light gray, micritic, with disseminated galena, appears to be at same elevation as unit 1, separated from it by unit 3, base at water level	6±	
1.	Limestone breccia, with galena and chert; limestone, gray, dense, base at water level	6±	

Samples from the mines and from Oronogo Circle were dissolved in 20-25 percent acetic acid (v/v) by the method described by Collinson (1963). A heavy mineral separation method on acid residues using tetrabromoethane concentrated conodonts and metallic sulfides from which conodont specimens and fragments of

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[3] Nomenclature used is that of the Missouri Geological Survey.

TABLE 1.—List of conodonts recovered as acid residues. Number indicates sample number; number in parentheses indicates number of specimens.

Formation	Locality
	1294-27: Oronogo Circle
Warsaw	1. No specimens
	2. Fragments
	3. No specimens
	4. Fragments
	5. *Gnathodus texanus* (18) *Neoprioniodus* (1) Fragments
	6A. *G. texanus* (1) *Ligonodina* (2) *Neoprioniodus* (2) Fragments
	6B. *G. texanus* (1) Fragments
	6C-D. No specimens
	6E. Fragments
	6F. *Neoprioniodus* (1) Fragments
	6G. *G. texanus* (8) Fragments
	Blue Mound mine
"Reeds Spring"	1. *Gnathodus texanus* (3) *Hindeodella* (1) *Ligonodina* (1)
	2. *G. texanus* (22)
	3. *G. texanus* (15)
	4. *G. texanus* (3)
	4A. *G. texanus* (1) *Lonchondina* (1) Fragments
	West Side mine
"M-Bed"	5A-B. No specimens
	5C. *Gnathodus texanus* (1) Fragments
	5D. *G. texanus* (3) Fragments
	5E. *G. texanus* (2) Fragments
	5F. No specimens
	5G. *G. texanus* (1) Fragment
	5H-I. No specimens
	5J. *G. texanus* (3) Fragments
	5K. *G. texanus* (7) Fragments
	5L. *Icriodus* (1) (contaminant)
	5M. *G. texanus* (1) *Icriodus* (1) (contaminant) Fragments
	5N. Fragment
	5O. No specimens
	5P. Fragment
	8A. No specimens
	8B. Fragment
	8C. Fragments
	8D-F. No specimens
"J-Bed"	9. No specimens
	Webber mine
"M-Bed"	6A-D. No specimens
	6E. *Gnathodus texanus* (1)
	7. Fragment
	Jarrett mine
"M-Bed"	10A. Fragment
	10B. Fragments
	11A. No specimens
	11B. *Gnathodus texanus* (2) Fragments
	11C. No specimens
	Griffin mine
"Short Creek"	12A. Fragment
	12B. Fragments
"M-Bed"	12C. *Gnathodus texanus* (11) *Ligonodina* (3) *Lonchodina* (45) *Neoprioniodus* (1) Fragments
"J-Bed"	13A. *G. texanus* (22) *Hibbardella* (6) *Ligonodina* (3) *Lonchodina* (31) *Metalonchodina* (2) *Neoprioniodus* (3) Fragments
	13B. *G. texanus* (36) *Taphrognathus varians* *Hibbardella* (1) *Ligonodina* (7) *Lonchodina* (46) *Metalonchodina* (4) Fragments
	13C. No specimens
	John Beaver mine
"M-Bed"	14A. *Icriodus* (1) (contaminant) *Lonchodina* (1)
	14B-C.; 15. No specimens
"Sheet Ground"	16A. *Gnathodus texanus* (40) *Hindeodella* (5) *Lonchodina* (1) Fragments
"M-Bed"	17A. No specimens
	18A. *G. texanus* (1) *Spathognathodus pulcher* (1) *Ligonodina* (2) *Lonchodina* (1) Fragments
	Big John-K mine
"M-Bed"	19. *Gnathodus texanus* (2) *Lonchodina* (6) Fragments
	23-24. No specimens
	Grace B mine
"J-Bed"	20. No specimens
	21. *Gnathodus texanus* (19) *Taphrognathus varians* (2) *Hibbardella* (2) *Ligonodina* (3) *Lonchodina* (34) *Metalonchodina* (1) *Neoprioniodus* (1) Fragments
"J-Bed"	22. *G. texanus* (35)

TABLE 1.—List of conodonts recovered as acid residues. Number indicates sample number; number in parentheses indicates number of specimens *(concluded)*.

Formation	Locality	Formation	Locality
"G, H-Bed?"	*T. varians* (3)		Little Greenback mine
	Ligonodina (6)		(Tongaha Mining Co. & Tom Kiser)
	Lonchodina (31)		31. Sample skipped
	Metalonchodina (3)	"M-Bed"	32A-B. No specimens
	Neoprioniodus (3)		33. Fragments
	Fragments		34A. *Taphrognathus varians* (1)
	25. Sample skipped		34B-E. No specimens
"J-Bed"	26. Fragments		34F. *Gnathodus texanus* (1)
	27-29. No specimens		34G. No specimens
	30. *G. texanus* (1)		34H. *T. varians* (1)
	Icriodus (1) (contaminant)		34I-35. No specimens
	Fragments		

conodont-like material were hand-picked. The specimens recovered were unaltered in appearance and displayed no physical evidence of reworking.

Conodont specimens recovered were studied and compared with reference collections of the Kansas Geological Survey and the Missouri Geological Survey. Identifications to generic and in some cases to specific level are given in Table 1. Letter designations (Table 1) of beds follow lithologic identifications established by Fowler and Lyden (1932) and Fowler, *et al.* (1935). E. H. Hare and P. R. Dingess kindly marked letter-beds and formations on lease maps. Parts of Figure 1 and Table 1 were compiled from this information.

The conodont fauna recovered correlates with the *Gnathodus texanus* s.s.-*Taphrognathus* Zone which Collinson, *et al.* (1962) established in the type section of Mississippian rocks in the upper Mississippi River valley. Their zone coincides with the Keokuk Formation. They correlated the zone approximately with the lower part of the cuIIα Zone of western Europe. The upper limit of the zone (Collinson, *et al.*, 1962) is reported as marked by the lowest occurrence of *Apatognathus*? and by the lowest abundant occurrence of *Taphrognathus varians* Branson and Mehl. The lower zonal boundary is characterized by the lowermost limit of abundant occurrence of *Gnathodus texanus* s.s. The zone is characterized by relatively few species but a great abundance of *Gnathodus texanus* s.s. Roundy and rare to common occurrence of *Taphrognathus varians* as well as *Taphrognathus* n. sp.

Rexroad and Collinson (1965) in a study of faunas from the Keokuk, Warsaw, and Salem formations in Illinois verified that the zone is dominated numerically by only two species, *Gnathodus texanus* and *Taphrognathus varians*. In several sections of Keokuk Formation studied they reported a fauna dominated by *Gnathodus texanus*, with no *Taphrognathus varians*. The fauna of the *Gnathodus texanus-Taphrognathus* Zone is much like that of the fauna of the *Taphrognathus varians-Apatognathus*? Zone which coincides (Collinson, *et al.*, 1962) with the Warsaw and Salem formations. The latter zone is characterized by greater abundances of *Taphrognathus varians* and *Neoprioniodus tulensis* Pander. Differences in the abundance of specimens seemingly indicate useful biostratigraphic subdivisions.

A total of 538 identifiable specimens was recovered as residue material from the 35 locations sampled. Of this number 261 are *Gnathodus texanus*, 197 are *Lonchodina*, 12 *Neoprioniodus*, 1 *Spathognathodus pulcher* (Branson and Mehl), 11 *Taphrognathus varians*, and 56 others judged of less biostratigraphic value. By numeric abundance of *Gnathodus texanus* over other species, the *Gnathodus texanus-Taphrognathus* Zone is judged present. Occurrence of four specimens of *Icriodus* (a pre-Mississippian conodont) is thought to be contamination due to the laboratory procedure as a Devonian conodont residue project was near completion in the laboratory when this project commenced. Apparently, these specimens of *Icriodus* are from insufficiently cleaned screens or straining apparatus but the possibility remains that the specimens were reworked as part of a mixed fauna.

A similarity of faunas by mine locations is shown in Table 1. The Blue Mound and West Side samples from the northeastern part of the Picher Field in Kansas contained large specimens (>1.75 mm) of *Gnathodus texanus* in a fauna which excluded *Taphrognathus varians*. Seemingly, this would be an early part of the zone. Samples from the Grace B and Griffin mines in the northwestern part of the field in Kansas contained a fauna also dominated by large specimens of *Gnathodus texanus* but which contained a few specimens of *Taphrognathus varians*. Probably this fauna indicates a later part of the zone. The John Beaver, Little Greenback, and other mines sampled contained a fauna judged insufficient for proper evaluation.

Specimens of ***Gnathodus texanus*** from Oronogo Circle were relatively small in size (<1.0 mm) compared with those found in all the underground locations. The Geologic Map of Missouri (McCracken, 1961) shows the location of the Oronogo Circle to be in an area of exposures of the Warsaw, Salem, and St. Louis formations. Identification of the meager fauna is inconclusive. Tentatively, a late portion of the range of ***Gnathodus texanus*** is indicated.

Thompson (1965) reported the ***Gnathodus texanus-Taphrognathus*** Zone present in two outcrops (SE NW sec. 1, T 35 S, R 25 E, and NE NE SW sec. 33, T 34 S, R 25 E) in southeastern Cherokee County, Kansas. The localities are geographically between the Picher Field and Oronogo Circle.

Only one zone of conodonts is presently recognized in the Tri-State District ore bodies. The Reeds Spring Formation (Table 1), because of its pre-Keokuk stratigraphic assignment by Moore (Moore, ***et al.***, 1951), should have contained an older fauna than the ***Gnathodus texanus-Taphrognathus*** Zone. No early Mississippian conodonts were found in the Tri-State ore bodies sampled. However, early Mississippian conodont faunas from outcrops in southwestern Missouri have been reported (Thompson 1967).

Conodonts occur in and adjacent to the ore bodies of the Tri-State District in sufficient numbers and variety of specimens to establish the presence of the ***Gnathodus texanus-Taphrognathus*** Zone. This is the first report of these microfossils in the underground mined area. Conodonts are preserved, apparently unaltered by mineralizing solutions, in mineralized bodies of rock. Faunas of conodonts may be useful as guides to age correlation of mineralized beds. Although this study is preliminary, sufficient information was gained to suggest that a similar study in other mineralized and semi-mineralized sedimentary rocks would be fruitful. Examination of exploratory cores for conodonts in areas in the Midcontinent in Mississippian rocks where traces of metallic sulfide are known is recommended. Such regional investigation of occurrence of traces of galena and sphalerite in cores containing faunas of conodonts of similar age as those in rocks of the Tri-State District possibly could point to favorable areas for future exploration.

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CLAYTON M. CROSIER[1]
RONALD G. HARDY[2]

Cellular Concrete of Kansas Pozzolanic Materials

ABSTRACT

Nearly 200 batches of cellular concrete, foamed, were made of Kansas pozzolans with portland cement and were autoclaved. Volcanic ashes produced concretes having unusually high strength-density ratios. The desirable conditions of making such concretes were broadly determined. The compressive-strength-versus-dry-density relationships of the concretes that satisfied these conditions were about 1500 psi at 40 pcf to 3000 psi at 55 pcf. The moduli of elasticity agreed closely with those given by the 1963 ACI Code formula.

For a number of years the State Geological Survey of Kansas has sponsored research in the Concrete Laboratory at the University of Kansas on the use of Kansas pozzolans, especially volcanic ashes, in cellular concrete.[2]

Nineteen different Kansas pozzolanic materials have been tried, ten have shown practical potential. These ten materials, their chemical analyses and their geographic sources are shown in Table 1. Nearly two hundred (195) batches of cellular concrete have been made. These have involved many variations: in materials, in proportions of the materials, in mixing, in curing, and in drying preparatory to testing. All were the foamed type of cellular concrete, some by the use of preformed foam, most by the mix-foaming method, all using the same commercial foaming agent. Nearly all specimens were autoclaved, usually at about 360° F. The resulting concretes have been tested or measured with respect to density, compressive strength and modulus of elasticity, the specimens being 3 x 6-in. cylinders, and volumetric stability by standard bars. In some cases flexural strength has been determined, and a few thermal conductivity tests have been made.

Analyses of the data show conclusively that the use of Kansas volcanic ash as the pozzolan yields cellular concrete of unusually high strength: 2000 psi at 45 pcf dry density, and 3000 psi at 55 pcf. The modulus of elasticity of this concrete is very nearly that given by the formula in the 1963 American Concrete Institute (ACI) Standard Building Code (1963), which in the notation of the present report is:

$$E = 33\ D^{1.5}\ f'^{0.5}$$

in which

E is the modulus of elasticity, psi,
D is the dry density, pcf, and
f' is the compressive strength, psi.

The flexural strength of this high-compressive-strength concrete has not been determined. Its fire-protection value is questionable—exposure to moderately rapid changes in temperature above 150°F, or perhaps to temperatures over 150 to 200°F without changes, usually produced craze-cracking, but quantitative determination of the extent and nature of this weakness will require further research.

The compressive-strength-versus-density relationships of 65 different batches of concrete made in this investigation are presented in Figure 1. The most significant data on these 65 batches is given in Table 2. In all but three of these concretes the pozzolan used was Kansas volcanic ash. The strength-density relationship is very nearly linear in the density range of Figure 1 and is represented conservatively by the equation:

$$f' = 115\ D - 3100.$$

At the often-referred-to density of 40 pcf the mean strength is nearly 1500 psi; at 45 pcf it is over 2100 psi, and at 55 pcf it exceeds 3000 psi.

That these high strengths are attributable primarily, perhaps solely, to the pozzolanic materials used is indicated by the dashed line and the three non-volcanic-ash points in Figure 1. The dashed line represents the linear mean of the highest strength-versus-density values of National Bureau of Standards (NBS) experimental concretes, as reported by Valore (1954, p. 821)

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[1] Associate Professor of Civil Engineering, University of Kansas; has conducted this research project since 1954.

[2] Chief, Mineral Resources Section, Kansas State Geological Survey; initiated this research in 1953.

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TABLE 1.—Properties and sources of pozzolans used.

Reference code	Kind of material*	Chemical analyses†, %						Location of bed or deposit in Kansas		Thickness of bed, ft
		SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO & MgO	K_2O & Na_2O	Land survey description	County	
LV-2	Volcanic ash, Pearlette	72.3	12.6	1.7	0.3	0.9	8.4	S ½, S 27, T 13S, R 10W	Lincoln	6.5
LV-1	Volcanic ash, Pearlette	72.5	11.6	1.2	0.5	0.8	6.6	same	Lincoln	6.0
GTV-3	Volcanic ash, Pearlette	72.8	11.7	1.8	0.2	0.6	9.0	NW ¼, S 24, T 30S, R 35W	Grant	15.0
GTV-1	Volcanic ash, Pearlette	72.8	12.3	1.5‡		1.0	8.1	NE ¼, S 1, T 30S, R 36W	Grant	23.5
RWV-1	Volcanic ash, Pearlette	72.8	12.1	1.7	0.2	0.9	8.7	NW ¼, S 14, T 3S, R 35W	Rawlins	14.0
NNV-1	Volcanic ash, Calvert	72.7	11.2	2.0	0.4	1.5	7.8	SW ¼, S 25, T 2S, R 22W	Norton	11.5
PRV-1	Volcanic ash, Pearlette	72.5	12.0	1.7	0.4	0.9	8.3	SW ¼, S 21, T 27S, R 12W	Pratt	14.0
ROV-1	Volcanic ash, Pearlette	74.1	11.2	1.9	1.0	1.4	7.7	SE ¼, S 14, T 25S, R 8W	Reno	10.0
RV-2	Volcanic ash, Pearlette	72.8	12.1	1.6	0.2	0.7	8.6	NW ¼, S 2, T 15S, R 11W	Russell	7.0
FR-3	Expanded shale stack dust	61.0	20.8	8.1	0.8	2.2	4.2	NW ¼, S 23, T 17S, R 19E	Franklin	25.0
Fc	Fly ash	46.1	21.8	20.1	1.6	2.6	3.3	commercial product		

* For additional information, see Kansas Geol. Survey Bull. 91 for FR-3, and 96-1 for all volcanic ashes.

† No other compound constituted as much as **one percent except that SO_3 was 1.1 percent of Fc, the fly ash.**

‡ Sum of Fe_2O_3 and TiO_2.

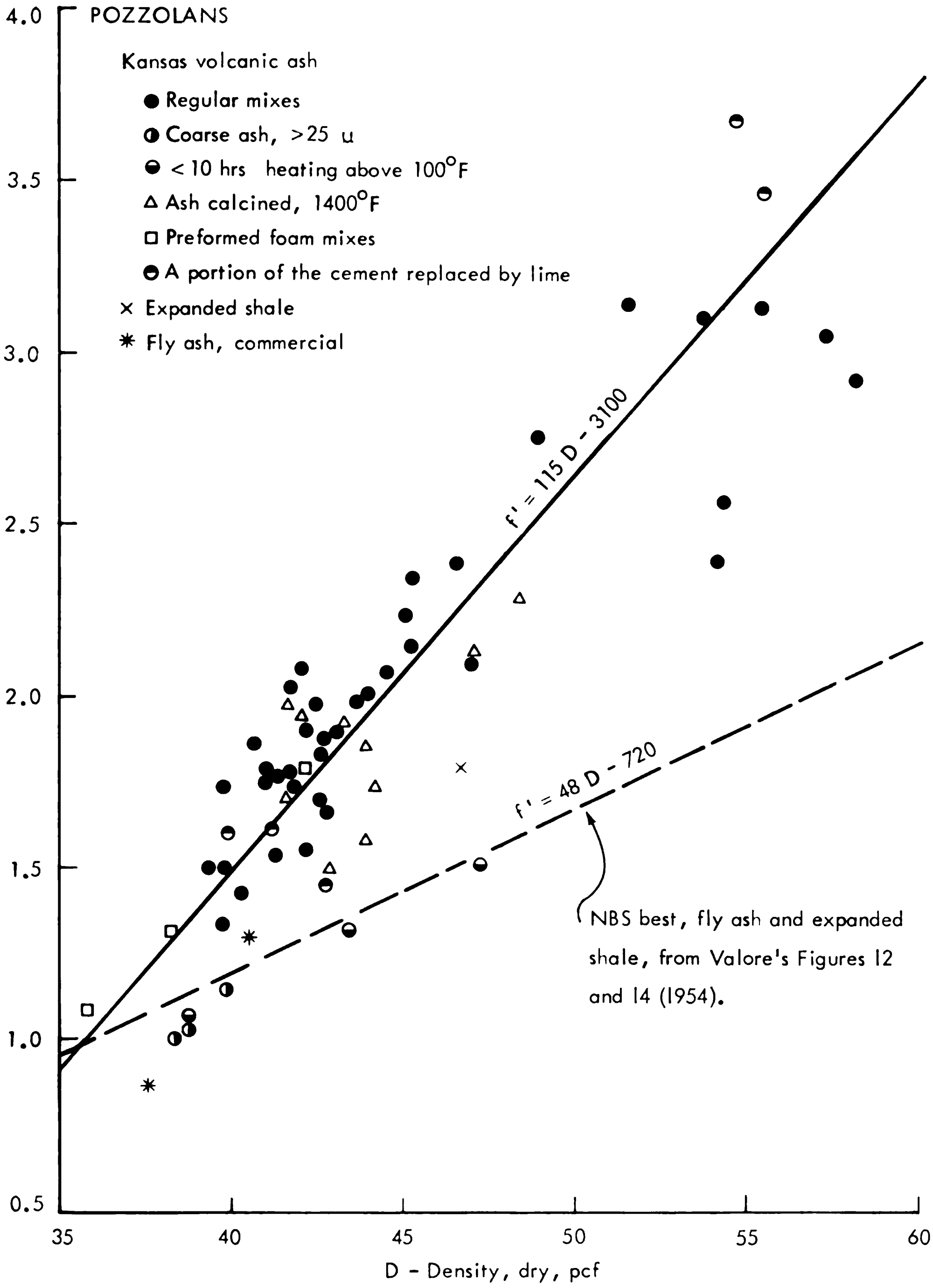

FIGURE 1.—Strength versus density of foamed cellular concrete made of type III portland cement and Kansas pozzolans.

TABLE 2.—Composition, density, strength and elasticity of the cellular concrete.

* C in this column indicates that the ash was calcined at 1400°F.
† Median diameter of particle, microns, determined by standard pipette test.
‡ P—Pre-formed foam used; all other batches were foamed by high-speed mixing; M—Moist cured for *x* days, all other batches moist cured 1 to 3 days; L-*b*—Lime, hydrated, was added to the portland cement, *b* being the ratio of lime to cement-plus-lime, by weight.
§ First number is hours heating from 100°F to full heat temperature, second number is hours at full heat ± ¼.
¶ Moisture content of the concrete when tested, percent.
nt = no test.

Batch ref. no.	Pozzolan				Water/Solids by weight	Special conditions‡	Autoclaved			Drying temp., max. °F	Density		M. C. @ T¶	Comp. strength			Elasticity		
	Kind (see Table 1)*	% Passing sieve no. 200	% Passing sieve no. 325	Median diameter†			Full heat °F	Hours Up to	Hours At§		Dry, pcf	$\bar{v}$%		n	f', psi	$\bar{v}$%	n	E, ksi	$\bar{v}$%
LV17	LV-2	99	94+	16.6	0.67	....	365	4	8—	135	41.2	1.3	1+	3	1530	5.8	3	371	5
LV19	" C	"	"	"	"	M-9	365	4½	8—	135	41.6	0.3	1—	2	1705	1.6	2	362	2
LV21	"	"	"	"	"	....	360	4+	8	135	43.8	0.1	1	2	1995	1.2	2	427	4
LV1B	"	"	"	"	"	....	365	3½	8+	140	42.4	1.1	1—	4	1975	2.5	4	425	3—
CLL1	"	"	"	"	"	L-.25	365	3+	8—	"	41.1	0.2	1—	4	1605	2.6	2	362	3—
CLL2	"	"	"	"	0.76	L-.50	365	3+	8—	"	39.8	0.8	1—	4	1595	7.2	2	319	1—
D3L1	LV-1	99+	96—	15.0	0.67	....	360	4	7½	"	45.2	0.2	1	4	2135	8.2	2	509	4—
D3L2	"	"	"	"	"	....	"	4	8—	"	45.2	0.1	"	4	2340	1.3	2	535	1+
B3L1	"	"	"	"	"	....	"	4	8—	"	46.9	0.2	"	4	2100	0.8	2	512	1—
A3L1	"	"	"	"	"	....	"	4+	7½	"	45.0	0.2	"	4	2230	3.6	2	500	0
LV23	"	"	"	"	"	....	"	4—	8—	"	44.5	0.2	"	3	2070	2.0	..	nt	..
LV24	"	"	"	"	0.68	....	"	4½	7+	"	41.7	0.3	"	3	1725	9.7	..	nt	..
F3L1	"	"	"	"	"	....	365	4—	7½	"	43.6	0.3	0	5	1985	3.4	2	405	1+
E3L1	"	"	"	"	"	....	"	4—	8—	"	40.7	0.1	0	4	1860	3.0	2	370	1—
C3L1	"	"	"	"	"	....	"	4—	7½	"	41.6	0.4	0	5	2015	2.1	2	424	2
GV1	GTV-3	100	98	18.6	0.58—	P M-149	357	5	6½	130	38.2	0.5	2	4	1315	6.1	..	nt	..
GV3	"	100—	97+	15.9	0.67	....	355	4+	7+	140	41.4	0.5	"	2	1770	1.4	2	405	0.2
GV4	"	"	"	"	"	....	"	4+	7+	135	42.1	1.7	3	3	1545	2.1	3	383	4+
GV5	"	"	"	"	"	....	"	4½	5+	"	38.7	0.8	3	3	1060	2.0	3	300	3—
GV6	"	"	"	"	"	....	"	"	"	"	43.3	0.7	1	3	1330	3.6	3	388	5—
GV7	"	"	"	"	"	....	"	"	"	"	47.0	1.0	2	3	1505	3.1	3	432	4
GV9	"	"	"	"	"	....	365	4½	8—	"	39.4	0.4	1	2	1500	0.8	2	345	4+
GV10	"	"	"	"	"	....	360	4+	8	"	42.1	0.2	"	3	1910	2.7	3	422	0.5
GV13	GTV-1	99+	96+	16.6	0.67	....	"	3	8+	140	42.7	0.7	"	2	1665	4.1	2	377	0
GV15	"	"	"	"	0.68—	M-2-9	365	4+	7+	140	39.8	0.6	"	4	1340	2.0	2	321	2+
RwV1	RWV-1	98+	94+	16.4	0.58	P M-151	358	4+	8—	130	42.2	1.0	2	4	1790	6.7	..	nt	..
RwV22	"	100	98	13.8	0.67	M-10	365	4½	8—	135	41.0	0.4	1	2	1790	1.9	2	382	2
" 24	"	"	"	"	"	....	360	4+	8	135	42.8	0.6	1	3	1875	2.9	3	454	2.4
RwVC2	RWV-C	100	98+	13.6	0.64	....	355	4½	7+	135	43.8	0.9	2	3	1580	8.6	3	450	3+
" 3	" "	"	"	"	"	....	"	4½	7+	"	44.1	0.9	2+	3	1740	10.0	3	448	2.5
" 4	" "	"	"	"	0.67	....	"	"	"	"	42.8	2.0	2	3	1495	18.5	3	425	2.5

" 5	" "	"	"	"	"	....	"	"	"	"	46.9	1.3	2	3	2125	0.9	3	498	1+
" 6	" "	"	"	"	"	....	"	"	"	"	48.3	1.2	2	3	2285	4.3	3	523	2
" 8	" "	"	"	"	"	M-9	365	4½	8—	"	41.6	0.5	1	2	1965	3.2	2	450	3+
" 9	" "	"	"	"	"	....	360	4	8+	"	43.8	0.5	1	3	1855	11.0	3	489	2.4
PV1	PRV-1	nt	nt	15—	0.67	....	365	3½	8	140	42.6	0.3	1	5	1830	1.6	3	415	2+
PVC1	" C	"	"	14.7	"	....	"	"	"	"	43.1	0.3	"	5	1910	4.4	3	438	3—
PV6	"	99	91—	14.1	0.68	....	"	4+	7—	"	40.3	0.0	"	2	1420	4.0	..	nt	..
E3P1	"	"	"	"	"	....	"	4+	7	"	42.6	0.5	"	5	1680	2.8	2	414	1—
F3P1	"	"	"	"	"	....	"	4+	8—	"	41.1	0.2	"	5	1765	5.6	2	391	2+
A3sP1	"	"	"	"	"	....	"	3+	8—	"	39.7	0.4—	"	5	1720	7.0	2	371	1
A3P1	"	"	"	"	"	....	"	3+	8—	"	39.7	0.1	"	5	1495	6.4	2	364	1—
CLP1	"	nt	nt	15—	0.74	M-6 L-0.50	"	3½	8	"	42.7	....	"	5	1445	6.2	3	378	3
NV2	NNV-1	100—	98+	13.4	0.67	....	365	3+	8	140	41.0	0.2	2	5	1805	7.6	3	436	0.8
NV3	"	"	"	"	"	....	360	4—	8—	140	42.0	0.2	1	3	2070	1.9	..	nt	..
NVC1	" C	"	"	13.3	0.67	....	365	3+	8	140	42.0	1.0	2	5	1925	2.2	3	427	1.2
RV1	ROV-1	nt	nt	25.2	0.67	....	365	3	8	140	39.8	0.6	4	5	1140	5.7	3	289	2.1
RV2	"	"	"	"	"	....	"	3½	8	"	38.3	....	3	6	1005	3.5	3	249	0.4
RVC1	" C	"	"	25.3	"	....	"	3	8+	"	38.5	0.5	2	6	1025	7.6	3	290	6.9
RP2	RV-2	99+	96	nt	0.57	P M-141	358	4—	8—	130	35.8	0.5	2	4	1080	3.6	..	nt	..
B21	Fr-3	100	99+	10.5	0.67	....	360	4+	8½	140	46.6	0.2	1	4	1790	3.7	..	nt	..
A15	Fc	99+	98	nt	0.67	M-6	365	4½	8—	135	37.6	1.0	1—	2	865	2.9	2	340	0
A16	"	"	"	"	"	....	360	4	8+	135	40.5	0.3	1	3	1300	3.0	3	347	2.3
MG1	GTV-1	100	98	nt	0.68—	....	360	6½	5½	140	42.9	0.9	0	3	1890	6.5	2	448	0.4
" 2	"	"	"	"	0.74+	....	"	5	5½	"	46.4	1.5	"	3	2380	8.6	2	560	1.3
" 3	"	"	"	"	0.68—	....	"	5+	6—	"	48.8	0.4	"	3	2740	0.3	2	606	0.4
" 4	"	"	"	"	"	....	"	6	5+	"	53.6	0.2	"	3	3090	3.7	2	694	0.2
" 5	"	"	"	"	"	L-.10	"	6	5½	"	55.3	0.4	"	3	3460	2.1	2	710	4.4
" 6	"	"	"	"	"	....	"	6½	5+	"	51.4	0.8	"	2	3140	0.6	1	616	..
" 7	"	"	"	"	"	L-.10	"	6	5½	"	54.5	0.9	"	3	3680	2.6	2	739	2.0
" 8	"	"	"	"	"	....	"	5½	5½	"	57.2	1.7	"	2	3050	5.3	2	762	1.8
" 8-B+C	"	"	"	"	"	....	"	10—	3½	"	55.3	1.1	"	3	3130	2.6	..	nt	..
ML1	LV-1	100	98	nt	0.68—	....	360	6½	4+	140	58.0	1.4	0	3	2910	9.9	2	805	2.5
ML2	"	"	"	"	0.72—	....	"	6	5	"	54.2	1.5	0	3	2560	1.4	2	652	3.1
ML3	"	"	"	"	0.76	....	"	5½	5+	"	54.1	0.6	"	3	2390	2.2	1	705	..

in his figures 12 and 14. These NBS concretes were made with either fly ash or expanded shale dust as the pozzolan. The fly ash and expanded shale concretes made in this research at the University of Kansas are in good agreement with NBS concretes. This is indicated by the three non-volcanic-ash points in Figure 1. (The strength of the expanded shale concrete was abnormally high because of the extreme fineness of the shale in that batch.) In all significant respects except for the pozzolan, these three concretes were made and tested like the other 62 in Figure 1.

Figure 2 shows clearly that the modulus of elasticity of this Kansas cellular concrete is in remarkably close agreement with the previously cited formula given in the ACI Building Code (ACI, 1963). This formula was based principally on research on concretes having densities in the range of 90 to 155 pcf (Pauw, 1963). That the modulus of these concretes having densities under 60 pcf, most less than 45, agree closely with the ACI formula is for this reason doubly significant.

Numerous factors besides the constituent materials and the density affect the strength and other properties of cellular concrete. Many of these have been studied in this investigation. Analysis of the data obtained, although as yet incomplete, shows that when Kansas volcanic ash is the pozzolan several factors produce effects that are different from those produced by the same factors on fly ash or expanded shale concrete. The selection of the 65 batches of concrete included in this study of strength versus density was made by applying the following *limitations* to the fifteen stated factors.

1. Cement materials used were:
   a. type III portland cement plus;
   b. calcium chloride of weight between 1.5 and 3 percent of the weight of portland cement; and
   c. either no lime or a weight of lime not greater than that of the cement.
2. The pozzolans were ground to fineness measured by a median diameter less than 17 μ or by the sum of the percentages passing no. 200 and no. 325 screens being not less than 195 percent.
3. The ratio by weight of portland-cement-plus-lime to pozzolan was 2 to 3, 0.67.
4. All test specimens were cylinders:
   a. that were nominally 3 x 6-in. in size; and
   b. that had height-diameter ratios between 1.9 and 2.0.
5. Specimens were cured by:
   a. moist storage one day in the molds and zero to nine days in the fog room followed immediately by
   b. autoclaving:
      (1) the full-heat temperature being 360 ± 5°F
      (2) this temperature being maintained for about 8 hours, limits being 7.25 to 8.75 at full heat, or
      (3) the heating period—the sum of the hours from 100°F to the end of the full-heat period—being not less than 11.5 hours
      (4) the cooling period being about 12 hours, the consequent entire cycle about 24 hours.
6. Specimens were dried to constant weight:
   a. at 130 to 140°F;
   b. with only gradual changes in temperature.
7. The moisture content of specimens at the time of compressive loading was not more than 4 percent.

Exceptions were made to each of four *limitations* for the following groups of batches (Table 2), each of which satisfied all but the one *limitation* indicated by number:

2. RVI, RV2, RVC1—The ROV-1 ash in these batches was at least 50 percent coarser than the ashes in the other concretes having the same densities—the strengths averaged 25 percent lower.

5.a. Batches RP2, GV1 and RwV1 were stored in the fog room about 150 days, then autoclaved. A specific study indicated that, for Kansas volcanic ash concretes, 90 to 180 days of such storage usually affects the autoclaved strength less than 2 percent.

5.b. (2&3). Cylinders of GV5, GV6 and GV7 were autoclaved by heating over 100°F for only 9¾ hours, at full heat (only 355°F) for only 5¼ hours. As Figure 1 shows, strengths were lowered about 15 percent at 39 pcf to 35 percent at 47 pcf.

5.b. (2&3). All MG batches were autoclaved at full heat for only about 5½ hours, but the heating period was at least 11 hours in all but one case. The strengths agree well with the linear mean curve of Figure 1. For the ML batches the times at full heat were somewhat less, the strengths were markedly less. Obviously, more data is needed on these higher density cellular concretes.

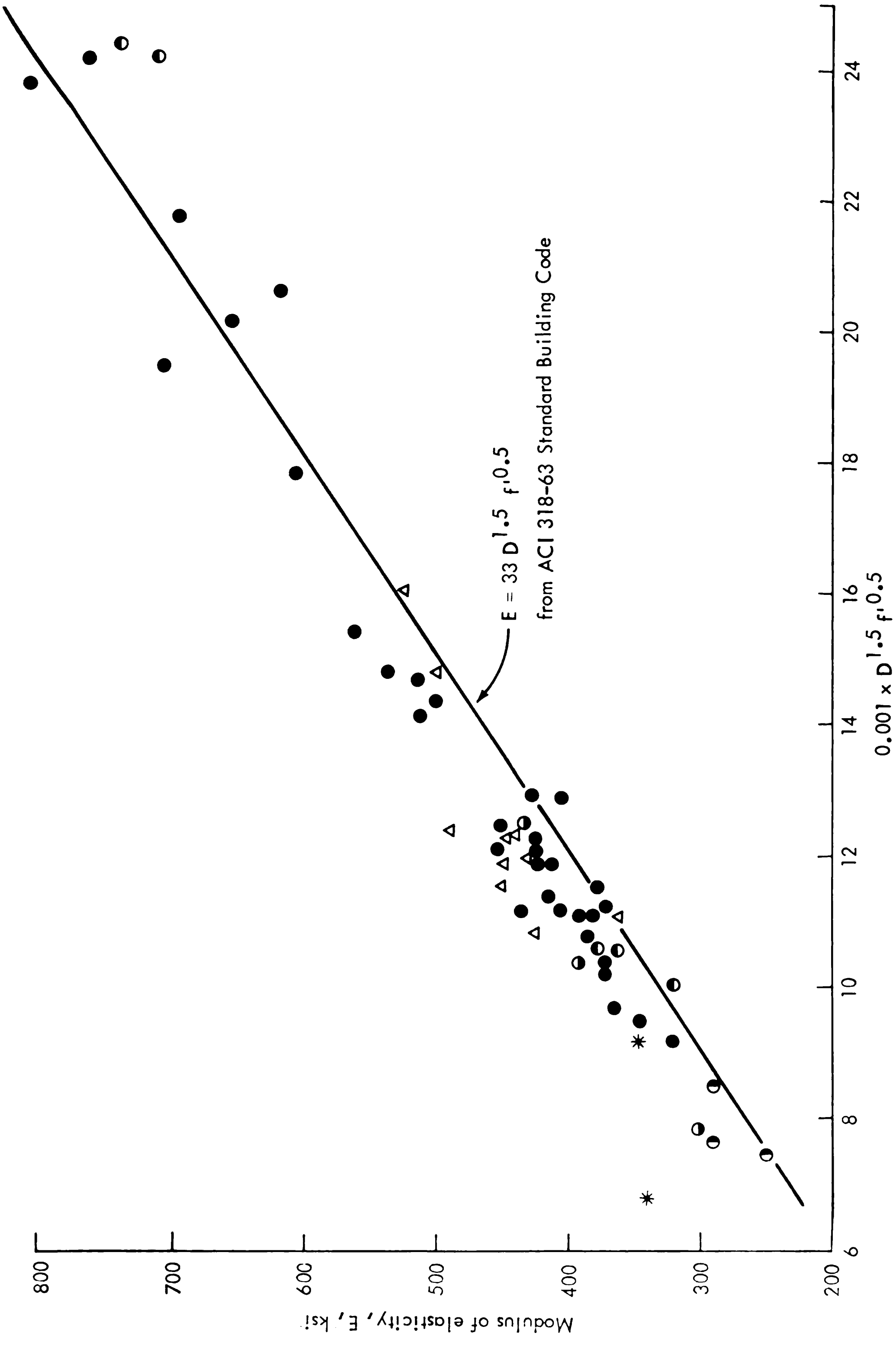

FIGURE 2.—Modulus of elasticity of cellular concrete made with Kansas volcanic ash.

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