THE PRODUCTION OF A LIGHTWEIGHT CONCRETE AGGREGATE
BY THE ACTION OF HEAT ON CLAY SHALES

by

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TABLE OF CONTENTS

LIST OF TABLES AND FIGURES

LIST OF TABLES AND FIGURES
(Cont'd.)

I. INTRODUCTION

Lightweight aggregate for concrete is a general term applied to materials which when mixed with cement and water produce a concrete weighing approximately one hundred pounds per cubic foot or less. Lightweight aggregates may be divided into three classes according to their source. These classes are: (1) Natural; (2) By-product; (3) Processed. Natural aggregates include such material as pumice, scoria, breccia, tuff and volcanic ash, all of volcanic origin. By-product aggregates include cinders, air-cooled slag, coke breeze, sawdust and grog (broken and cull ceramic materials). The third class of lightweight aggregates, the processed aggregates, can be further subdivided into two classes; the expanded slags and the expanded shales. Of all of the lightweight aggregates mentioned, the expanded shales seem to offer the most possibilities in Virginia because of the wide occurrence of shale deposits.

In the past decade, there has been a growing tendency for power and industrial plants to substitute modern pulverized coal boilers for the conventional stoker-fed boilers. This has been of great concern to producers of lightweight concrete products who use cinders as an aggregate for their

products. The pulverized fuel boilers produce a very fine fly ash which has not been shown to be suitable as a concrete aggregate. Therefore, it can be assumed that the sources of supply of suitable cinders will diminish. In Virginia, there are no known naturally occurring materials available and steel mill slag could not be used because of prohibitive transportation costs. On the other hand, there is available an unlimited supply of easily quarried shale.

Cinder Block Inc. of Roanoke, Virginia, is aware of the possible shortage of cinders and are on the lookout for a suitable substitute. The company has purchased a tract of land on which is located a fairly large deposit of clay shale. Preliminary investigation of this shale has indicated that the shale will expand or bloat to a cellular, lightweight product when exposed to a high temperature, but other information concerning the shale is desired.

The purpose of this investigation is to determine the temperature at which bloating of the shale occurs; whether a better product can be obtained by prolonged heating of the shale; whether the effect of heat on shale from various locations on the tract is the same; the effect of the chemical composition on bloating; the effect of rapid heating on bloating; and whether the bloated shale will produce a high-grade lightweight concrete.

II. LITERATURE REVIEW

Structure of Silicates

Early Investigation.- In 1910, Pukall[24] wrote of his
experiments with clay substances. He prepared silicates of
various metals by fusing silica and sodium carbonate and then
precipitating the silicates by double decomposition with met-
al salts and sodium silicate. An aluminum silicate with the
ratio of 3 parts of silica to 2 parts of alumina to 6 parts
of water was produced. Pukall proposed the following struc-
tures for kaolin acid, kaolin (which he termed dehydrated ka-
olin acid) and the sodium salt of the acid.

$$\begin{array}{ccc}
OH \cdot SiO_2 \cdot Al(OH)_2 & OH \cdot SiO_2(OH)Al \searrow & OH \cdot SiO_2 \cdot Al(OH)NaO \\
\parallel & \qquad\qquad O & \parallel \\
OH \cdot SiO_2 \cdot Al(OH)_2 & OH \cdot SiO_2(OH)Al \nearrow & OH \cdot SiO_2 \cdot Al(OH)NaO \\
\\
\textit{KAOLIN ACID} & \textit{KAOLIN} & \textit{SODIUM SALT}
\end{array}$$
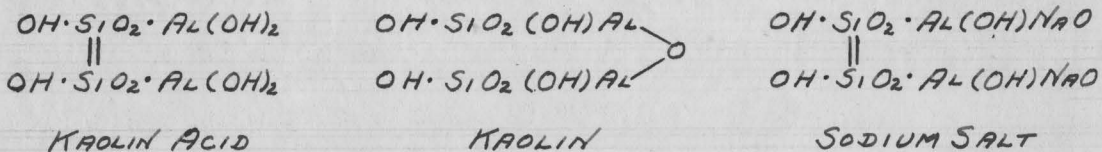
Singer,[30] in 1911, proposed the structural formula
$(HO.Si.O.OAl(OH))_2O$ for kaolin. He stated that only metasi-
licic acid may be considered the foundation of all natural
and artificial silicates. All possible pyrosilicic acids are
derived from $O:Si(OH)_2$ (metasilicic acid). Singer criti-
cized Pukall's formula unfavorably and pointed out that a
double silicon bond, like a double carbon bond, is a source
of weakness in a compound rather than one of strength.

Considerable criticism of Pukall's formula was made by Wegscheider and Manchot.[32] Wegscheider considered complex silicates as salts of polysilicic acids in which the Si groups are linked through oxygen. He made reference to Pukall's formula and pointed out that Manchot had criticized Pukall by stating that combinations between Si atoms in SiO combinations take place only in reducing conditions and silicates are formed without reduction. In inorganic combinations between silicon atoms the products are unstable, especially those containing oxygen. A formula with directly combined silicon atoms would not explain the stable condition of kaolin. If silicon atoms were directly combined in silicates, the oxygen atoms must also be directly combined. It is known that compounds with directly combined oxygen atoms are unstable (viz H_2O_2). Wegscheider went on to state that regarding silicates as salts of polysilicic acid does not prevent one from regarding those containing aluminum as salts of alumino-silicic acid with oxygen combinations between the silicon and aluminum.

Hexite and Pentite Theory.- In 1913, W. Asch and D. Asch[4] proposed what they termed the "Hexite and Pentite" theory. They theorized that if five and six molecules of $Si(OH)_4$, respectively, unite together, splitting off water and retaining the quadrivalency of the silicon so as to form

a closed ring, the following structural formulae are pro-
duced:
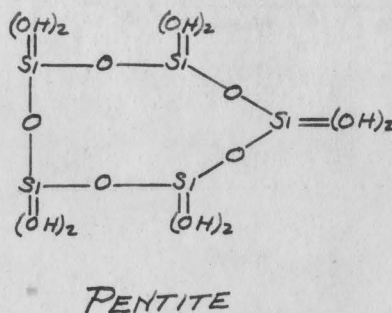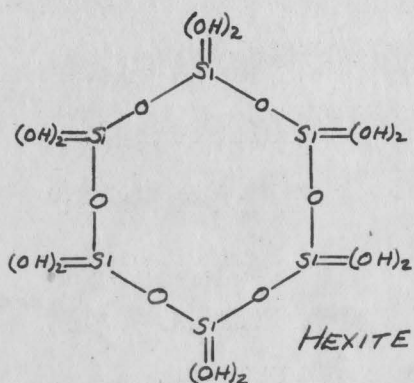
HEXITE

PENTITE

Also, by substituting aluminum atoms for the silicon atoms,
aluminum hexite and pentite are formed. Thus, for example,
$3H_2O.3Al_2O_3$ and $6H_2O.6SiO_2$ would yield the formula:
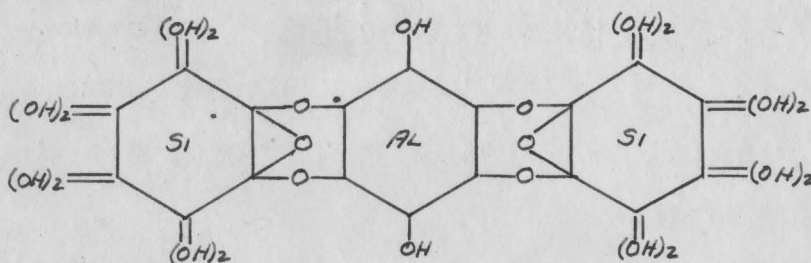
The Asch brothers show, by various combinations of alum-
inum and silicon hexites and pentites, how the structure of
a large number of known silicates can be explained.

X-ray Study of Silicates.- In spite of the large amount
of work done and the apparent logic of some of the theories
mentioned, it remained for Bragg and Pauling[20] to elucidate
the different problems of the structure of the silicates by

X-ray analysis. They showed that the difference between different classes of silicates is directly dependent on the silicon and oxygen arrangement and only indirectly dependent on the remainder of the structure. Occasionally the silicon oxygen structure forms a closed group like an ordinary acid radical, but usually the structure is endless and extends continuously throughout a given crystal. The silicon atom in the silicate is always associated with four oxygen atoms, tetrahedrally arranged with regard to the silicon. The way in which these tetrahedral groups are related determines the structure of the particular silicate.

Mellor[20] states that many silicates are derived from various silicic acids. Metasilicic acid is formed by the following reaction:

$$Na_2SiO_3 + 2HCl \rightleftharpoons H_2SiO_3 + 2NaCl$$

Metasilicic acid exists as hydrogel and hydrosol; the hydrogel being the gelatinous form and the hydrosol being a solution. If sufficiently dilute, all of the metasilicic acid takes the hydrosol form. Clear solutions containing 5% colloidal silica can be obtained by dialysis. These solutions can be concentrated in a flask by boiling until they contain about 14% H_2SiO_3, which, on standing a few days, transform to a hydrogel. The transformation of hydrosol to hydrogel is retarded by small amounts of hydrochloric acid or an alkali

hydroxide and accelerated by sodium carbonate. If the clear solution is evaporated in a vacuum at 15° C., clear, transparent jelly is obtained which, when dried over sulphuric acid, has the approximate composition of $SiO_2.H_2O$ or H_2SiO_3 - metasilicic acid. This also may be prepared by dehydrating the gelatinous silicic acid with 95% alcohol. When the gelatinous silicic acid is dehydrated with ether and dried, an acid of the composition $SiO_2.2H_2O$ or H_4SiO_4 - orthosilicic acid - is obtained. Orthosilicic acid loses water on exposure to air. On the other hand, silica gel of the formula $SiO_2.nH_2O$ has a remarkable capacity for absorbing water from the air.

For chemical purposes, silicates have been classified on the basis of these silicic acids[20]. Thus, willemite, Zn_2SiO_4 corresponds to the orthosilicic acid as does olivine, $(MgFe)_2SiO_4$; zircon, $ZrSiO_4$; and tephroite, Fe_2SiO_4; wollastonite, $CaSiO_3$ and enstatite, $MgSiO_3$ correspond to the metasilicic acid.

Another series of silicic acids result when two molecules of orthosilicic acid condense into one of ortho-di-silicic acid, $H_6Si_2O_7$. This gives rise to a series of ortho-di-silicates such as serpentine, $MgSi_2O_7$; barysilite, $PbSiO_7$, etc. In a like manner, the condensation of two molecules of metasilicic acid gives rise to a series of meta-di-sili-

cates. This process is continued for ortho-, meta-, tri-, and poly-silicic acids.

Tetrahedra Structure of Silicates.- W. L. Bragg[10] was the first to show that silicates consist of essentially the relatively larger oxygen ions in close packed arrangement, either cubic or hexagonal. The ions are held together by strongly charged metallic ions which fit into the spaces between them. In tetrahedral spaces are to be found the smallest and most highly charged ions, Si^{++++}, Be^{++}, Ti^{++++}, Al^{+++}, Mg^{++}, Fe^{++}, etc. Larger ions, such as Ca^{++}, Na^{+}, and K^{+}, introduce distortion into the structure. The symmetry of the particular silicate adjusts itself to fit these ions with minimum distortion, with the result that the unit crystal cells are large and complicated because of low symmetry.

Bragg made the following conclusions in regard to the structure of the silicates:

(a) Whatever the silicon to oxygen ratio, silicon is always situated within the tetrahedral group of four oxygen atoms, which is very constant in form from crystal to crystal.

(b) The structures are typical coordination structures - four oxygens at the corners of a tetrahedron, six of an octahedron, etc. The whole structure may be regarded as a fabric of which these groups are the stitches, the groups being

joined together by sharing oxygen atoms.

(c) The coordination numbers among silicates are $Be^{++}4$; $B^{+++}3,4$; $Na^{++}6,8$; $Mg^{++}4,6,8$; $Al^{+++}4,5,6$; $Si^{++}4$; $Ca^{++}6,7,8$; $Sc^{+++}6$; $Ti^{++}6$; $Mn^{++}4,6,8$; $Fe^{++}4,6,8$; $Fe^{+++}6$; $Zn^{++}8$; $Ba^{++}6,12$; $K^{+}6,8$.

Diopside is a typical meta-silicate of the formula $CaMg(SiO_3)_2$. Each silicon atom is surrounded tetrahedrally by four oxygen atoms. Two of these atoms are common to neighboring groups and two are not. The tetrahedra are linked by their corners into endless chains parallel to the C axis of this monoclinic crystal. It is incidental that the SiO_4 groups are cemented sideways by calcium and magnesium ions.

Tetrahedral Groups.- In orthosilicate groups[20] exemplified by olivine, $(MgFe)_2SiO_4$ and zircon, $ZrSiO_4$, the tetrahedral groups are independent like quadrivalent acid radicals. The oxygen atoms each have one negative charge, the oxygens being attached to the silicon by a single valency. These negative charges attract the metallic ions of the crystal. This structure may be represented by the following diagram:
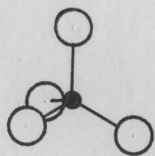
○ OXYGEN
● SILICON

When two of the above groups are linked by their common corners, that is, through two groups sharing one atom of oxygen, a grouping Si_2O_7 results of a valency of 6 (since the linking oxygen atom now has no charge).
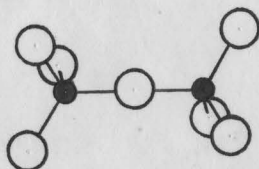
Other groups have been observed in which three tetrahedra (C) are united into a ring through sharing of three oxygen atoms thus forming a group Si_3O_9, whose effective valence is again 6. An example of this structure is the rare mineral bentonite, $BaTiSi_3O_9$. Similarly, rings of six tetrahedra have been found, (E). An example of this structure is beryl, $Be_3Al_2Si_6O_{18}$. A ring composed of four tetrahedra (D) has also been detected in certain zeolites.
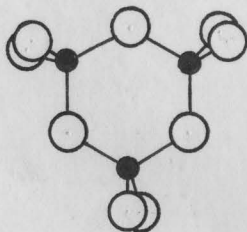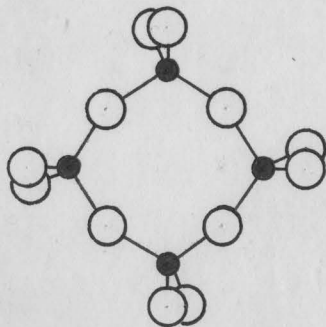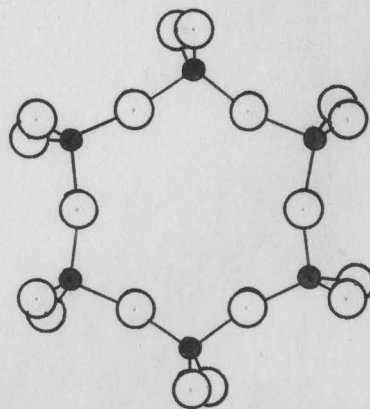
| C | D | E |

The silicon and oxygen tetrahedra are also found in
chain, band, and sheet types of structure. An example of the
chain type (F) is pyroxene in which the tetrahedra are joined
corner to corner in a row which extends indefinitely in both
directions.
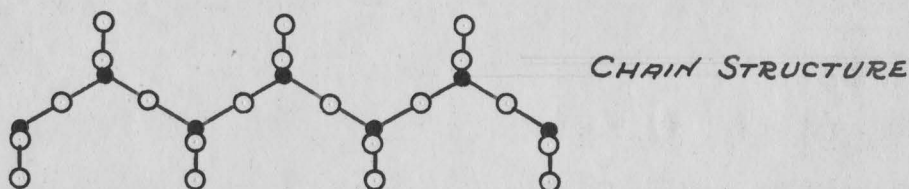
CHAIN STRUCTURE

Examples of the band type of structure exist in the am-
phiboles in which two chains are bound together by sharing of
oxygen atoms. Chains and bands are fibrous as in asbestos.

In a like manner, sheets are formed by linking three
corners of the tetrahedra, leaving only one free oxygen. Ex-
amples of this structure are mica and talc.

SHEET STRUCTURE
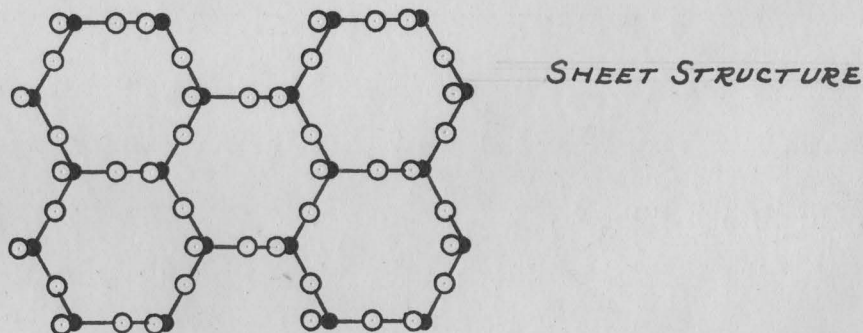
If three dimensional networks are formed, the tetrahe
are linked by all four corners so that there are no oxygen
atoms left carrying charges to attract the positive ions.
The result is one of the forms of silica (e.g. quartz). In
most of the structures already considered, a certain number
of silicon atoms may be replaced by aluminum. Whenever this
occurs, each tetrahedral unit of the framework acquires a
resultant negative charge on account of the lower valency
(fewer outer ring electrons) of aluminum. Metallic ions are
then incorporated into the interstices of the structure.
The felspars are examples of this arrangement.

The silicates thus, for the most part, form crystals
which are not to be thought of as loose aggregations of sim-
ple molecules, but as giant molecules in which the whole
mass is bound by chemical linkages which extend to the lim-
its of the crystal. The infusibility and insolubility of
the silicates is presumably the result of this structure.

The Action of Heat on Clays and Shales

Melting Points of Silicates.- In 1908, Rieke[26] investigated the fusibility of various lime-alumina-silica mixtures of the following compositions:

(1) Al_2O_3-SiO_2-0.1 to 12CaO   (2) Al_2O_3-2SiO_2-0.1 to 12CaO   (3) Al_2O_3-3SiO_2-0.1 to 12CaO.

Cones were made and the melting point taken at the temperature at which the point of the cone first touched the bottom of the furnace. The main point of interest, as far as results were concerned, seemed to be that the melting point decreased as the ratio of lime was increased up to a certain point. When this point was reached the increase in the lime ratio increased the melting point.

Dittler,[11] in 1911, continued the work on the melting point of the silicates. He investigated artificial diopside which he found had a freezing interval between 1290° C. and 1250° C. He also investigated other silicates including labradorite which showed a freezing interval from 1200° C. to 1160° C. He concluded that two processes occur in silicate fusion. Firstly, the transformation from the crystal to the isotropic amorphous state which is not accompanied by a noticeable heat reaction and secondly, the liquefaction of the amorphous mass which is accompanied by a heat reaction.

Temperature - Specific Gravity Relationship.- Knote,[17] in 1911, did some work on relating the temperature of the clays to the specific gravity. His results show that the specific gravity of the clay he was investigating increased to a temperature of 450° C. and then began decreasing until 650° C. was reached. At this temperature, it began increasing again and continued to increase to 1000° C., which was the highest temperature it reached.

Dehydration Temperatures.- Further study by Wallach,[31] in 1913, revealed that when clays were heated slowly from 20° C. to 1150° C. a sudden arrest in the heating curve took place at 100° C., presumably due to loosely held water being driven off. The second break occurred at 500° C. He concluded that dehydration was invariably complete at 600° C. A slow liberation of heat between 900° and 1000° C. was assumed to indicate the formation of an aluminate.

Rieke's[25] work showed that a similarity exists between kaolin and high-grade fire clay in that both show a distinct temperature lag at 500° C. and a longer halting period at from 560° to 580° C. He found that some ferroginous clays behave similarly to fire clays while others show practically no halting point. The loss of chemical water begins only at 450° C. The dissociation velocity increases rapidly with the temperature. By heating sufficiently long, practically all

of the water may be expelled between 400° and 500° C. The last portions of the chemical water are given off with diffi-culty.

Searle[29] expressed his thoughts on the subject of the effects of heat on clays, probably from the experience of others as he does not mention having done any experimental work. He stated that (1) mechanically combined water is driv-en off at 100° C.; (2) as the temperature is raised chemical decomposition occurs; and (3) at 500° to 800° C. a quick evo-lution of water occurs. These conclusions are more or less in agreement with other investigators, but the basis for his sec-ond observation is not shown.

Boudouard and Lefranc[7] further corroborated the temper-atures at which water is given off in the heating of clays, and found that hygroscopic water is evolved continuously and slowly to 450° C. Liberation of combined water started at 450° C. and was complete at 500° C. Exception to this was noted in the case of a halloysite which showed further loss at 800° C., and one of the clays also showed the same loss. They investigated seven clays and two halloysites.

Expansion on Heating.- Braesco[8] measured the expan-sion and contraction of test pieces of clay as they were heat-ed to 1000° C. by means of a telescopic apparatus located on the exterior of the furnace and through which he could view

the test pieces as they were heated in the furnace. In all cases, there was first expansion followed by contraction. The temperature at which contraction began for glauconite was 590° C. This temperature, according to him, shows no relation to the temperature of dehydration of the aluminum silicates.

Bigot, [6] in 1921, concluded that clays and kaolins expand before reaching their fusion temperature but bauxites do not. Mixtures of kaolin and silica show very marked swelling before fusion. This property may be utilized in preparing artificial pumice stone by rapidly heating schist and porcelain to their swelling temperatures.

Evolution of Heat.- Wohlin, [34] in 1914, attacked the problem from the standpoint of the temperature at which exothermic or endothermic reactions took place. He used 29 different "clays, bauxites and allied materials". He increased the temperature of his furnace to 710° C. during the first 25 minutes and from there to 1300° C. at the rate of 12° C. per minute for a total heating time of 75 minutes. He found that (1) from 560° C. to 580° C. clays showed heat absorption; (2) exactly at 960° C. (at this rate of heating) heat was evolved more or less vigorously the longer the period of heat absorption; and (3) bauxite of the formula $Al_2O_3 \cdot H_2O$ showed heat absorption at 540° C., while bauxite of the formula $Al_2O_3 \cdot 3H_2O$ showed this at 310° C. Both varieties often show

at 1060° C. a considerable evolution of heat due to a change
in the form of Al_2O_3. Wohlin states that this does not occur
in the case of $Al_2O_3 \cdot 2H_2O$ at 1060° C. and, because of this
phenomenon, one can distinguish $Al_2O_3 \cdot SiO_2 \cdot 2H_2O$, $Al_2O_3 \cdot 4H_2O$
and $Al_2O_3 \cdot 3H_2O$ when present in sufficient amounts.

Houldsworth and Cobb,[15] in 1925, observed that the
changes responsible for the exothermic effects observed at
950° C. with clays could be completed on continued heating
at 900° C. and that occurring with Al_2O_3 at 1060° C. could
be completed at 1000° C. These changes in clays and in pure
Al_2O_3 are distinct and different phenomena. None of the in-
dications of exothermic and endothermic reactions with clays
can be attributed to the presence of small amounts of impur-
ities and their possible interaction with the clay. No evo-
lution of heat at 950° C. was found when mixtures of Al_2O_3
and SiO_2 of the ratio $Al_2O_3 : 2SiO_2$ were heated.

Other Theories on Action of Heat.- Mellor and Scott,[21]
in 1925, reached the conclusion that the dehydration of kao-
linite does not take place at a fixed temperature and is com-
pleted at temperatures above 500° C. At these temperatures
they state that kaolinite decomposes into free silica, free
alumina and water. The exothermic break in the heating curve
about 900° C. is connected with a transformation in the form
of Al_2O_3. Sillimanite, similar to the natural mineral, can

develop below 1200° C. and probably above that temperature forms solid solutions with $3Al_2O_3.2SiO_2$. Above 1700° C., $3Al_2O_3.2SiO_2$ may be formed.

The research staff of the General Electric Company,[3] in 1926, attempted to follow by X-ray methods the 500° C. to 900° C. changes in kaolinite. They found that kaolinite completely breaks down at about 600° C., forming a silicate which is stable to about 900° C. The silicate presumably is $Al_2O_3.2SiO_2$. At higher temperatures mullite is formed. The X-ray patterns of natural sillimanite and mullite prepared by heating andalusite at 1550° C. for five hours are different. The pattern of the mullite agrees with those obtained from fired clay mats. The mineral formed in clays at 1000° C. is mullite and not sillimanite. The formation of mullite occurs in all clays examined by heating them five hours at 1050° C. The temperature at which complete stability is reached varies with the clay.

Causes of Bloating or Swelling of Clays and Shales

Shale.- Orton and Staley[23] at the Ohio State Universi-
ty, in 1908, burned shale containing 5.42% Fe, 2.87% C. and
3% S. to a number of temperatures and under three sets of
conditions. The conditions were: (1) rapid oxidation and
normal vitrification (2) slow oxidation followed by normal
vitrification and overfiring and (3) normal oxidation and
vitrification followed by heavy reduction without increase
in temperature and closing by raising temperature and main-
taining reducing conditions. Their conclusions follow.
Iron in clays tends to oxidize to Fe_2O_3 in the absence of
carbon or sulphur. The formation of ferrous silicate does
not seem to cause the evolution of gases. Sulphur in any form
may give rise to gases at high temperatures by oxidation, dis-
sociation or replacement by SiO_2. Since in the unoxidized
portion of clay, carbon remains longest, the content of sul-
phur will be less reduced by the action of heat and subse-
quently the sulphur will cause the evolution of more gas,
causing swelling. Hence sulphur is the most important cause
of bloating. Clays high in carbon and iron but low in sulphur
blacken by improper burning but do not swell. Clays low in
iron but high in carbon and sulphur swell without blackening.
Clays high in sulphur containing neither iron nor carbon

swell on the exterior first.

Blistering of Cornish Stone.- Calcott,[9] in 1912, investigated the "blistering and bubbling of Cornish Stone". He said that this fault is found only in mixtures of buff and purple stone and is probably due to the unequal fusibility of the two, giving rise to the evolution of gases from the buff stone (the less fusible of the two) after surface vitrification due to the purple stone.

Bennet,[5] in 1941, made the following observations on bloating of Cornish Stone. He said that bloating is due to the expansion of entrapped air when ware is viscous as well as to the decomposition of gas-forming material. Softer fluxes cause bloating more than harder ones. Fineness of grinding has little effect on the bloating tendency.

Lightweight Aggregate.- Harper,[14] in writing of the construction of ships with lightweight aggregate during the first World War, said, "It was decided to imitate the volcanic rock by baking a clay into a clinker in a kiln. - - - A clay containing vegetable matter was selected. - - - At a temperature of about 2000° F. the vegetable matter turns to a gas inflating the clinker, leaving disconnected isolated cells when cool."

Prevention of Bloating.- Kallauner,[16] in 1930, gave as
the cause of bloating in ceramic products, when heated, the
presence of enclosed gases, i.e., water, air, oxygen, carbon
dioxide and sulphur dioxide. He said to prevent bloating:
(1) the raw materials should be washed (2) excessive fatness
removed (3) carbonaceous substances burned out at 500° C.
(4) firing should progress slowly (5) the steam in the kiln
space should be reduced to a minimum (6) sufficient draft
should be provided (7) firing at 800° to 900° C. must be car-
ried out in a reducing atmosphere to decompose sulphates (8)
the access of air should be facilitated so that carbonaceous
substances may be burned.

Shuen,[27] in 1937, gave as the causes of "blistering in
ceramics" the entrainment of air in excessively fat or harsh
clay, the reaction of iron oxide and carbonaceous matter, or
decomposition of iron oxide at high temperatures, formation
or liberation of water at high temperatures or overburning.

Methods of Bloating Clays and Shales

Fuller[12] wrote that light, spongy or vesicular burned clays when used as concrete aggregate have the advantage of good strength with lightweight. Rapid firing in rotary kilns is probably the best burning process. Such burned products, to be suitable, should show a fairly uniform, fine, cellular structure rather than one with large and irregular cavities. The pieces should be light enough to float in a salt solution of specific gravity of 1.1.

Additives that Promote Bloating.- It has been stated[2] that good insulators for furnaces have been made by adding sawdust, coal and kieselguhr to clay and firing the mixture.

Organic materials such as cut grass, oats, or other cereals, tan bark, coke dust, wood shavings, sawdust and peat have been mixed with clay and fired for making porous products[1].

Meyer and Pukall[22] suggested procuring artificial porosity in ceramic bodies by a chemical reaction between either the moisture in the plastic mass or constituents of the clay and finely divided metals which are added. These reactions cause the evolution of gases. Metals experimented with were aluminum, calcium, magnesium and silicon.

Loginov[19] described the bloating of various Russian

clays. Clay cylinders 10-20 mm. in diameter were prepared
from shale crushed to 1/30 mm. The firing was done at
1250° C. Swelling and clinkering occurred at 1150° C. to
1180° C. Various additions to the clay were tried, includ-
ing gas coal 2%, oil shale 5%, and swamp ore 4%. Swamp ore
gave the best results, producing a product weighing
0.56 gm./cc.

In 1940, Loginov[18] also wrote of preparing the product
described but with a thin crust on the globules which is im-
permeable to water. He did this by adding about 3% iron ox-
ide and 2% combustible agent, such as coal or peat.

<u>Natural Bloating Clays or Shales.</u>- Whitlotch[33] de-
scribed the bloating characteristics of a West Tennessee
clay with the view of its use for lightweight ceramic prod-
ucts or lightweight aggregate. He said that the clay fires
naturally to exceptionally low densities. Between cones 1
and 2 the apparent specific gravity ranges from 1.29 to 1.58
and the apparent porosity ranges around 40%. Additions to
the clay of 10-25% lignitic clay gave products of 1.09 and
0.91 apparent specific gravities or 60 to 57 lb. per cu. ft.

Patents

Early Patents.- The first United States patent
(#235,961) found pertaining to porous clay was issued to one
Dr. Georg Praetorius,[49] of Breslau, Prussia, Germany, on De-
cember 28, 1880. His invention was the preparation of porous
earthenware for use in "filtering, absorption and various
kinds of chemical treatment". He prepared the porous earth-
enware by mixing iron and lime free clay with powdered char-
coal, fluorspar and feldspar, shaping it to the desired form
and firing it in a kiln. The charcoal burned out, resulting
in the porous earthenware.

U. S. Patent #248,094, dated October 11, 1881, was is-
sued to Charles Gilman,[40] of Eldora, Iowa. His invention was
a process for making a porous, earthenware building material.
Clay, resinous sawdust and water were mixed thoroughly in a
mill until plastic. The plastic mass was then extruded
through iron or steel pipe from eight to twelve inches in di-
ameter to form "logs" about six to twelve feet long. These
logs were fired on a "slow steadily increasing fire" until
the "vapors" and steam were driven off. The temperature was
then raised rapidly until nearly white hot. This consumed
the sawdust and brought the clay to the first stages of vit-
rification. On cooling, the logs could be sawed into "planks",

bored, grooved, planed or carved with edged tools.

To William Lenderoth,[46] of Ontario, Canada, was issued
U. S. Patent #426,642 on April 29, 1890. His process was essentially the same as the foregoing patents except he claimed
greater strength by the addition of sand to the clay. Also,
he saturated the sawdust or other combustible matter with water before mixing.

The Hayde Patent.- Probably the most important patent
issued for this type of material was to Stephen J. Hayde[42],
February 12, 1918, U. S. Patent #1,255,878. In his patent,
Hayde specifically stated that clay or shale which was not
suitable for making brick could be used even if it contained
lime forming materials or sand. He suggested crushing the
material to about four inches and then burning it in a "rotary or other type" kiln at a temperature above 1500° F. -
preferably from 1700° to 2200° F. - for about two hours.
The clinker formed is allowed to cool in the air slowly until it will not disintegrate when sprayed with water. He
then suggests immersing the clinker in water to hydrate any
lime that might have been formed. The clinker is then
crushed and sized and mixed with cement or other hydraulic
material, molded into the desired form and allowed to harden.

Recent Related Patents.- In 1918, U. S. Patent
#1,314,752 was issued to O. Olsen[47]. This was a patent

whereby a mixture of coarse and fine pieces of shale, which have been burned but not clinkered, is used for an aggregate with Portland cement to produce a light concrete or may be used like fuller's earth for refining sugar or oil.

A British patent, #132,247, was issued to Olsen[48] about the same time which specified shale for use in concrete after being sintered or subjected to a temperature of 2000° F., or over, without thoroughly melting.

B. J. Fallon[39] was issued a patent (#1,528,759) on March 10, 1925. His process was for forming porous aggregate for use in making light concrete or other articles. A mass of clay is first fired in a kiln until a temperature of 800° F. is reached. The supply of air is then reduced and steam is passed through the bed of burning fuel into the clay to produce a reducing action thereon. Air is then again supplied to the clay to cause the mass to expand and thereby produce a porous material.

Frank A. Glass[41] secured U. S. Patent #1,536,482 on March 19, 1925, for a process of producing a hard, cellular clay product. His process consisted of mixing clay or shale with finely divided fuel and burning the mass on a grate by forcing air up through the mass to support combustion.

On May 4, 1926, U. S. Patent #1,583,521 was issued to

Carl William Boynton[38] for a process of making vesicular products by subjecting shales or clays high in iron, sulphur, sodium and potassium to heat which swells them. The expanded clay or shale is then crushed and screened for use as a lightweight concrete aggregate.

Bohlig[37] obtained Swiss Patent #126,755 in 1927 for making argillaceous clinker by preparing thick layers of calcined clay mixed with bituminous dust and burning the mass to clinker.

Charles E. Kraus[44] was issued U. S. Patent #1,741,574 on December 31, 1929, for a "method of forming cellular bodies which consists in heating a mixture of clay, bentonite, carbonaceous material and flux sufficiently to form sealed cells".

U. S. Patent #1,818,101 was issued to Kemper Slidell[51] on August 11, 1931, for a cellular or bloated clay product. In the process, granular material is fed into a heat zone maintained at bloating temperatures. The material while in granular form and in the heat zone is subjected to fusion while confined. The bloated material is extruded under pressure due to its expansion.

Tajamul Husain[43] obtained U. S. Patent #1,897,667 on February 14, 1933, for a cellular fired clay product suitable for fireproofing building block. The product is prepared by generating and releasing a gas in a clay slip of fluid con-

sistency by the interaction of an acid and a carbonate added
to the slip.

U. S. Patent #1,903,821 was issued to Sherman Q. Lee [45]
on April 18, 1933, for a process for bloating earthy materi-
als. In the process, a liquid containing molasses and fer-
rous sulphate in solution is added to a mass of earthy ma-
terial which is then heated to first evaporate the solvent
portion of the liquid and then to cause bloating by fusing
the earthy materials and liberating gases through the mass.

U. S. Patent #2,112,380, issued on March 29, 1938, to
C. M. Price, [50] describes a process in which siliceous ma-
terials having the desired porosity (or attaining it owing
to the presence of carbonaceous matter) are mixed with commi-
nuted marl of higher melting point and the mixture is burned
in a rotary kiln until the comminuted marl coats the sur-
face of the siliceous materials and seals the pores.

III. EXPERIMENTAL

Purpose of Study

The purpose of this study is to investigate the feasibility of producing a cellular lightweight concrete aggregate by heating clay shale until it reaches the point of incipient fusion. At this stage of heating, a bloating occurs in the shale caused by the release of gases. This results in a material having a cellular structure and a lower bulk density. In this study, an attempt will be made to determine the optimum temperature required to produce bloating of the shale; the effect of the chemical composition on the bloating of the shale; the bloating characteristics of different shale samples taken from different locations on the Ferguson Property in Roanoke, Virginia; the effect of the length of time to which a shale is exposed to a particular temperature on its bloating characteristics, and the comparison of the strength of concrete made with bloated shale as an aggregate with the strength of concrete made with cinder as an aggregate.

Plan of Investigation

Literature Review.- A search of the literature was made
for the purpose of ascertaining what had been done on the use
of bloated shale as a concrete aggregate and to become famil-
iar with the methods used by others.

Sampling.- All shale samples were obtained from a plot
of land which will hereafter be referred to as the Ferguson
Property in Roanoke. The samples were obtained from core
drillings of eight test holes. The location of the Ferguson
Property and the test holes are shown in Fig. 1.

Bloating Characteristics.- Samples from various levels
in each test hole were exposed to 2200° F. temperature in a
gas-fired muffle furnace. The degree of bloating was deter-
mined on each sample by determining the bulk density of the
sample before and after firing by means of a mercury volu-
meter.

Determination of the Bloating Temperature of Shale.-
For these tests, twelve samples of the shale were placed in
the furnace and the temperature increased to 2200° F. over a
period of one hour and thirty-six minutes. As the tempera-
ture reached each of the following points one sample was with-
drawn from the furnace and its bulk density determined:
1500° F., 1600° F., 1700° F., 1800° F., 1850° F., 1900° F.,

AVENUE

WEST

RORER

PATTERSON AVENUE

BOULEVARD

STREET — 20TH

21ST

22ND

STREET

BLOCK 39

TEST HOLE NO.4 O

400'

50'

TEST HOLE NO.3 O

525'

BLOCK 44

O TEST HOLE NO.2

100'

82.37'
O
TEST HOLE NO.8

50'

BLOCK 52

312'

186'

150'

BLOCK 53

TEST HOLE NO.6 O

TEST HOLE O NO.7

TEST HOLE NO. 5 O

BLOCK 38

BLOCK 45

X PURPLE
X BUFF
TEST HOLE NO.1 O

425'

BLOCK 51

400'

BLOCK 54

275'

50'
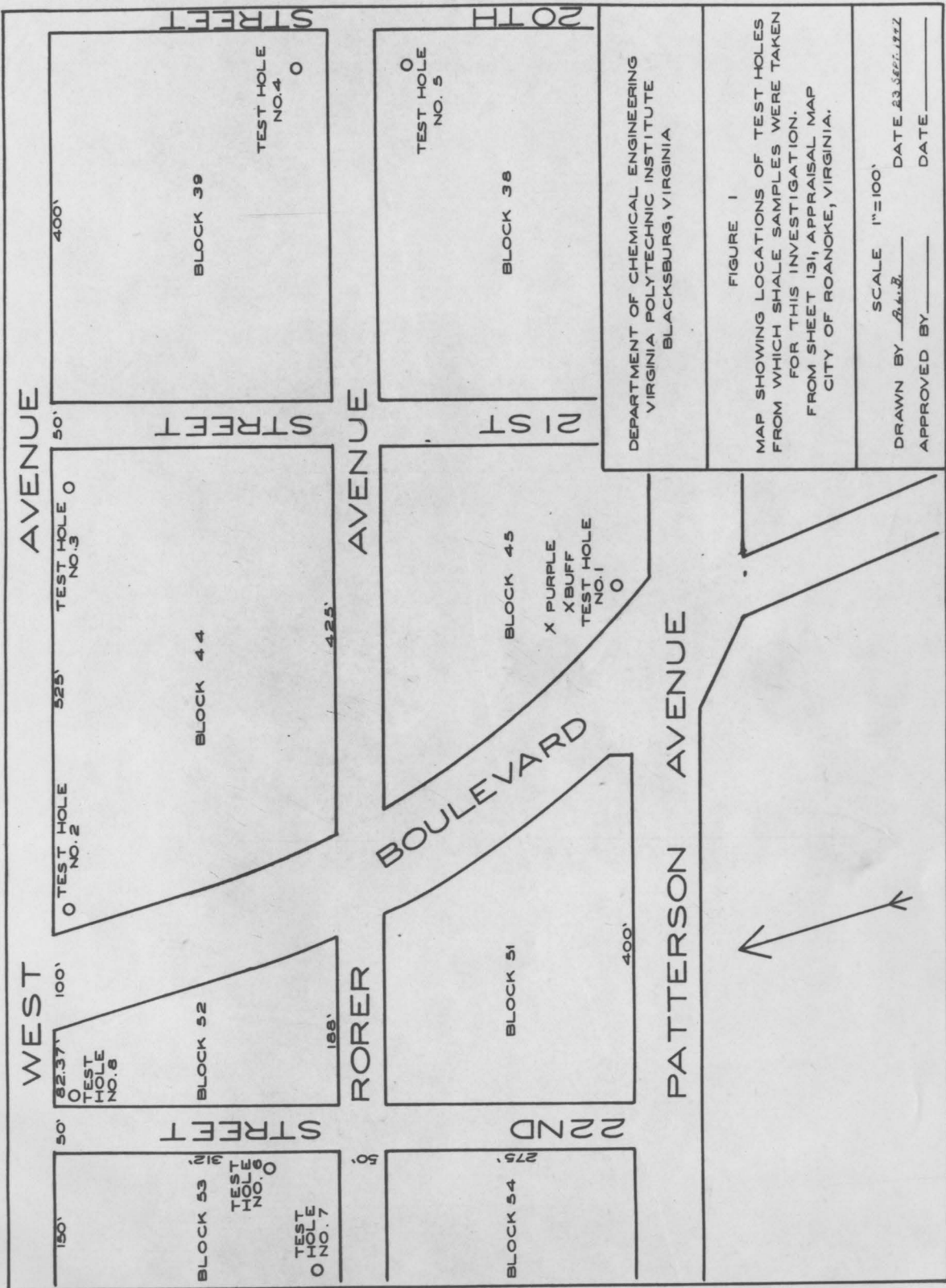
DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

FIGURE 1

MAP SHOWING LOCATIONS OF TEST HOLES
FROM WHICH SHALE SAMPLES WERE TAKEN
FOR THIS INVESTIGATION.
FROM SHEET 131, APPRAISAL MAP
CITY OF ROANOKE, VIRGINIA.

SCALE 1"=100'

DRAWN BY _B.H.B._   DATE _23 SEPT. 1977_

APPROVED BY _____   DATE _____

1950° F., 2000° F., 2050° F., 2100° F., 2150° F., and 2200°
F. In this way, the amount of bloating which had occurred
when the sample was removed from the furnace could be deter-
mined.

Effect of Prolonged Heating on the Bloating of Shale.-
To determine if better bloating could be obtained by expos-
ing the shale samples to a particular temperature for an ex-
tended period of time, fifteen shale samples were placed in
the muffle furnace and the temperature increased to a point
where bloating was known to occur (by previous tests). The
furnace was then adjusted so as to maintain this selected
temperature through the remainder of the tests. One sample
was removed each five minutes after the selected temperature
was reached and its bulk density determined. Thus, the ef-
fect on bloating of exposing the shale to a particular tem-
perature for extended periods could be determined.

Effect of Chemical Composition on Bloating.- The chemi-
cal composition of a poor bloating shale and of a good bloat-
ing shale was determined and the results compared in an at-
tempt to determine which of the elements or compounds pres-
ent is responsible for the bloating.

Strength of Bloated Shales as a Concrete Aggregate.-
Enough bloated shale was procured to make three concrete test
cylinders using the bloated shale as the aggregate. Three

additional cylinders were made using cinders as the aggregate.
All factors, including grading of aggregate, water-cement ra-
tio, method of molding, and method of curing were held con-
stant for all cylinders. Compressive strength tests were
then made on the cylinders to compare the strength of con-
crete produced from the bloated shale aggregate with that pro-
duced from cinder aggregate.

Materials

Shale Samples.- The samples of shale used in this in-
vestigation were all obtained from the Ferguson Property lo-
cated in West End, Roanoke, Virginia. Eight test holes were
core drilled on the property and samples of the cores were
taken at various levels to be used in the investigation. All
samples are identified by two numbers, the first number indi-
cating the test hole and the second indicating the depth at
which the sample was taken. Thus, sample #3-76 was taken from
hole number 3 at a depth of 76 feet. Fig. 1 shows the loca-
tion of the Ferguson Property in Roanoke and the location of
each test hole. Two other samples were taken from the face of
a road cut running through the property and are designated by
the notations "buff" and "purple" by reason of their colors.

The sources of these samples are also shown on the map.

Samples were taken at the following levels in the test holes:

Test Hole No. 1. Samples were taken at 9, 16, 26, 35, 45, 55 and 72 feet. Each sample weighed between one and three pounds and consisted of a cylinder 2½ inches in diameter. This test hole was 72 feet deep.

Test Hole No. 2. Samples were taken at 20, 25, 31, 38 and 42 feet. In this test hole the shale deposit was encountered at 20 feet and it extended to the 52.4 feet level, at which point drilling was discontinued.

Test Hole No. 3. Only two thin layers of shale were encountered when this hole was drilled. The first occurred at a depth of 30.9 feet and was 3 feet thick. The second layer was found at 75.2 feet and extended to 77.3 feet, at which point drilling was discontinued.

Test Hole No. 4. This hole was drilled to a depth of 67.1 feet without striking shale that would make an integral core. A sample of loose, crumbly shale was obtained from this hole but the depth from which it came could not be determined.

Test Hole No. 5. In drilling this hole, a series of shale layers were encountered. The first, at a depth of 5 feet, was 1.7 feet thick; the second, at a depth of 7.7 feet, was 0.6 feet thick; the third, at a depth of 10.2 feet, was

2.1 feet thick; and the last, at a depth of 16 feet, was 5 feet thick. Samples were taken at 6, 8, 10, 12 and 21 feet.

Test Hole No. 6. This hole, drilled in the bottom of a small valley, yielded nothing but a stiff red clay. Drilling was discontinued at 21 feet. Samples were taken at 11 and 21 feet.

Test Hole No. 7. While the shale in this test hole was soft and crumbled easily, it was found to be continuous from a depth of 5 feet to 45 feet. Drilling was discontinued at 45 feet. Samples were taken at 10, 18, 26, 33, 41 and 45 feet.

Test Hole No. 8. Shale was encountered at a depth of 2 feet at this location and continued to a depth of 42.2 feet, at which point drilling was discontinued. The cores obtained were excellent. Samples were taken at 5, 12, 22, 29 and 42 feet.

Two other shale samples of about one hundred pounds each were taken from the face of a road cut located on the property. These shales were relatively close together but differed considerably in appearance, one being a buff color and the other a purple color. Accordingly, one was designated as "buff" and the other as "purple".

Ceramic Blocks.- Ceramic blocks 6 inches long by 4 inches wide by 1 inch thick. Used in the furnace as platforms to place the samples on while being burned. Obtained from Cinder Block Inc. of Roanoke, the manufacturer being unknown.

Pyrofax Gas.- Used as fuel for the muffle furnace. Obtained from Natural Gas Distributors, Inc., Roanoke, Virginia.

Pyrometric Cones.- Research size, 1-1/8 in. long. Numbers 020 (1202° F.), 015 (1481° F.), 07 (1814° F.), 03 (2039° F.) and 11 (2417° F.). Manufactured by Edward Orton, Jr., Ceramic Foundation, Columbus, Ohio. Used to verify optical pyrometer and thermocouple temperature readings.

Analytical Chemicals

Ammonium Carbonate.- Powder, lot #38. Manufactured by the General Chemical Company, New York, N.Y. Used to flux the shale samples for the alkali determination.

Ammonium Chloride.- Lot #123141. Manufactured by the J. T. Baker Chemical Company, Phillipsburg, N.J. Used for the precipitation of the calcium in the alkali determination.

Ammonium Hydroxide.- Reagent grade of specific gravity 0.90, code 1239, lot #6133, having an NH_3 content of 28%. Manufactured by the General Chemical Company, New York, N.Y. Used for precipitating iron and aluminum hydroxide.

Ammonium Oxalate.- Lot #9340. Manufactured by the J.T. Baker Chemical Company, Phillipsburg, N.J. Used for the precipitation of calcium in the calcium determination.

Calcium Carbonate.- Lot #8243. Manufactured by the J.T. Baker Chemical Company, Phillipsburg, N.J. Used with ammonium chloride in sintering the sample for the alkali determination.

Disodium Hydrogen Phosphate.- Lot #7914, control WRVT. Manufactured by Mallinckrodt Chemical Works, Philadelphia, Pa. Used to precipitate magnesium in the magnesium determination.

Ferrous Ammonium Sulphate.- Lot #9340. Manufactured by the J.T. Baker Chemical Company, Phillipsburg, N.J. Used to standardize the potassium permanganate solution in the determination of iron.

Hydrochloric Acid.- 37% HCl, specific gravity of 1.187 at 60° F. Manufactured by the Grasselli Chemicals Department of the E.I. DuPont de Nemours and Company, Wilmington, Del.

Hydrofluoric Acid.- Lot #40715, 52 to 55% HF. Manufactured by Merck and Company, Inc., Rahway, N.J. Used in the determination of silica.

Methyl Orange.- Lot #1.11239. Manufactured by the J.T. Baker Chemical Company, Phillipsburg, N.J. Used as an indicator in the neutralization of the solution from which iron

and aluminum were precipitated as the hydroxides.

Potassium Bisulfate.- Lot #40647. Manufactured by
Merck and Company Inc., Rahway, N.J. Used to flux the iron
and aluminum oxides preparatory to the determination of iron.

Potassium Ferrocyanide.- Lot #29. Manufactured by the
General Chemical Company, New York, N.Y. Used as an indica-
tor to show the reduction of iron in the determination of
iron.

Potassium Permanganate.- Lot #82439. Manufactured by
the J.T. Baker Chemical Company, Phillipsburg, N.J. Used in
a 0.1 N. solution in the titration of iron.

Silver Nitrate.- Lot #1.1239. Manufactured by the J.T.
Baker Chemical Company, Phillipsburg, N.J. Used to indicate
the presence or freedom of chlorides in filtrate washings.

Sodium Carbonate.- Anhydrous, granular. Lot #1.1140.
Manufactured by the J.T. Baker Chemical Company, Phillips-
burg, N.J. Used in the fusion of the sample in the determi-
nation of silica.

Sulfuric Acid.- Lot #91946. Manufactured by the J.T.
Baker Chemical Company, Phillipsburg, N.J. Specific gravity
1.84; assay 96.8%.

Filter Paper.- #597 for fine precipitates and #595 for
gelatinous precipitates. 12½ cm. diameter. Manufactured by
Schleicher and Schull Co. Inc., New York, N.Y.

Apparatus

Muffle Furnace.- A gas-fired, six burner, muffle furnace having inside dimensions of 15 inches long by 12 inches wide by $7\frac{1}{2}$ inches high. Maximum operating temperature 2200° F. Model #16F oven furnace, serial #5662. Manufactured by the American Gas Furnace Company, Elizabeth, N.J. Used to heat shale samples.

Millivolt Meter.- Scale marked in degrees Fahrenheit. Range 0 to 3000° F. Model 269, #54523. Manufactured by the Weston Electric Instrument Company, Newark, N.J. Used in conjunction with thermocouple for temperature readings of muffle furnace.

Thermocouple.- An alumel-chromel thermocouple having a range to 2200° F. Manufactured by the Weston Electric Instrument Company, Newark, N.J. Used to measure temperature of muffle furnace.

Gas Meter.- Sargent Wet Test Gas Meter. Manufactured by the Precision Scientific Company, Chicago, Illinois. Used to calibrate rotameter.

Rotameter.- 12-3/4 inches long by $\frac{1}{2}$ inch O.D., scale marked 0 to 250 in centimeters. Manufactured by Schutte and Koerting Co., Philadelphia, Pa. Used to measure gas flow to muffle furnace.

Optical Pyrometer.- Disappearing filament type with potentiometer. Has two range scales, one from 1400° to 2200° F. and the other from 1950° to 3200° F. Model 8621. Manufactured by the Leeds and Northrup Co., Philadelphia, Pa. Used to measure temperature of muffle furnace.

Analytical Balance.- Capacity in each pan, 200 grams. Sensitivity, 1/10 milligram. Catalog #1-915. Manufactured by the Fisher Scientific Co., Pittsburgh, Pa. Used in weighing shale samples and in chemical analysis.

Analytical Balance Weights.- Serial #6520. Manufactured by William Ainsworth and Sons, Denver, Colorado. Purchased from the Fisher Scientific Co., Pittsburgh, Pa. Used in conjunction with analytical balance.

Crucibles, platinum.- Capacity 20 ml., 33 mm. in diameter, 33 mm. high, weight approximately 16 gms. with cover. Obtained from the Fisher Scientific Co., Pittsburgh, Pa. Used in silica determination.

Drying Oven.- Range from 35° to 180° C., 110 volts, 60 cycles, 5.5 amperes, 600 watts. Serial #100-2161; catalog #1250; type A. Designed and built by the Precision Scientific Company, Chicago, Ill. Used in chemical analysis.

Muffle Furnace.- Working temperature 1750° F.; maximum temperature 1850° F.; 220 volts; 6.13 amperes. Serial #1400, type 9921. Manufactured by the Cooley Electric Manufacturing

Co., Indianapolis, Ind. Used in heating shale samples.

Jaw Crusher.- Blake type with 6-inch corrugated jaws.
Manufactured by the Universal Crusher Co., Cedar Rapids,
Iowa. Used to reduce size of shale samples.

Air Compressor.- Nash Hytor Compressor, size A1574,
test #E-2006, RPM 3500, pressure 40 lbs./sq.in. Driven by
a three-phase, 200 volt, 60 cycle, 15 HP Western Electric in-
duction motor. Manufactured by the Nash Engineering Co.,
South Norwalk, Conn. Used as a source of air for the gas-
fired muffle furnace.

Scales.- Ohaus scales, 20 kilogram capacity, 22 in.
double beam. Manufactured by the Newark Scale Works, Newark,
N.J. Used to weigh large samples.

Volumeter.- Speil-Wilson[36] mercury balance volumeter
consisting of an Ohaus double beam balance of 20 kilogram
capacity and one gram sensitivity, to which is attached a
steel wire cage for immersing and weighing the sample in mer-
cury. Volumeter was assembled in the shop of the Industrial
Engineering Department and is now located in the Ceramics
Laboratory of Virginia Polytechnic Institute. Used to deter-
mine volume of shale samples.

Screens.- Tyler Standard Screens. Mesh numbers 3/8, 4,
8, 14, 28, 48, 100 and 200. Manufactured by the Tyler Screen
Co., Cleveland, Ohio. Used to grade cinders and bloated shale

in preparation of concrete test cylinders.

Testing Machine.- Hydraulic compression testing machine, capacity 400,000 lbs., located in the Materials Testing Laboratory of Virginia Polytechnic Institute. Manufactured by the Riehle Testing Machines Division of the American Machine and Metals Company, Inc., East Moline, Ill. Used to test concrete test cylinders.

Concrete Molds.- Cast iron, split molds, 6 inches in I.D. by 12 inches high. Manufactured by the Walker Machine and Foundry Co., Roanoke, Virginia. Used to mold concrete test cylinders.

Curing Autoclaves.- Cylindrical vessels 6½ feet in diameter, 60 feet long. Operating pressure 150 lbs./sq.in. Equipment owned by and installed at Cinder Block Inc. of Roanoke, Virginia. Manufactured by the McCarter Iron Works, Norristown, Pa. Used to cure concrete test cylinders.

Miscellaneous Glassware.- Beakers, flasks, burrettes, evaporating dishes, etc. Used in chemical analysis.

Method of Procedure

<u>Procedure for Determining the Bloating Characteristics
of Different Samples of Shales.-</u> Samples weighing approxi-
mately 25 grams were broken from the core drillings obtained
from the test holes in an iron mortar with a pestle. The sam-
ples were carefully cleaned of extraneous matter with a wire
brush. They were then weighed accurately on an analytical
balance. After being weighed in air, the samples were weighed
suspended in water. The densities of the samples were calcu-
lated by dividing the weights in air by the differences of the
weights in air and the weights in water.

The weighed samples were then placed on ceramic refrac-
tory slabs and placed in the furnace in such a position that
the gas flames would impinge directly on them; the furnace was
lighted and the temperature was raised from room temperature
to 2200° F. over a period of two hours in order to duplicate
firing schedules. Temperature readings were taken for every
50° F. rise in temperature and the time recorded. Up to a
temperature of 1500° F., the thermocouple alone was used to
indicate the temperature. Beginning at 1500° F., readings
were also taken with the optical pyrometer as a check. Stand-
ard pyrometric cones were used in each test to indicate tem-
peratures. These cones were 020 (1202° F.), 015 (1481° F.),

07 (1814° F.), 03 (2039° F.) and 11 (2417° F.). Thus, three
temperature indicating devices were used as a check on the
temperature.

The pyrofax gas was measured with a rotameter which was
calibrated by means of a Sargent Wet Test Gas Meter. The gas
flow was measured in order that duplication of the rate of
heating could be obtained in the several different tests.

When the temperature of the furnace had reached 2200° F.
the samples were removed from the furnace, cooled and weighed
in air. The bulk densities of the burned samples were deter-
mined by dividing the volume of the samples as determined on
the Speil-Wilson volumeter into the weight of the sample.
Since the bulk densities of the samples were known both be-
fore and after burning, a comparison of the amount of bloat-
ing for the various samples could be made.

Samples subjected to this test were each designated as
explained on page 33. The locations of the test holes are
shown in Fig. 1. The following samples were treated according
to the foregoing procedure:
1-9, 1-16, 1-26, 1-35, 1-45, 1-55, 1-72, 2-20, 2-25, 2-31,
2-38, 2-42, 2-50, 3-30, 3-76, 3-77, 4-67, 5-5, 5-6, 5-8, 5-10,
5-12, 5-15, 5-21, 6-11, 6-21, 7-10, 7-18, 7-26, 7-33, 7-41,
7-45, 8-5, 8-12, 8-22, 8-29, 8-42, buff and purple.

Procedure for Determining the Bloating Temperature of
Shale.- Three tests were made to determine the temperature
at which bloating occurred. In these three tests only one
type of shale was tested each time. The shale designated as
"buff" was used for the first test; shale #7-33 was used for
the second and the shale designated as "purple" was used for
the third test. Eighteen 25 gram samples were prepared and
weighed for these tests. The samples were numbered from 1
through 12 and the other six were designated A, B, C, D, E,
and F. Samples A, B and C were not fired but were used to de-
termine the bulk density of the shale by means of the mercury
volumeter. Samples D, E and F were weighed in air, dried to
constant weight, saturated with water by boiling for two
hours and standing in water for twelve hours, and weighed
suspended in water. From the data on samples A, B, C, D, E,
and F, the amount of moisture in the samples as received was
determined; the maximum water absorption of the shale was de-
termined; and the bulk density of the samples was determined.

The remaining twelve samples were placed in the furnace
on individual refractory slabs. The furnace was then adjust-
ed so as to reach a temperature of 2200° F. in two hours.
Samples were removed from the furnace as the furnace reached
the indicated temperatures:

Sample Number	Removed at $^\circ$ F.	Sample Number	Removed at $^\circ$ F.
1	1500	6	1900
2	1600	7	1950
3	1700	8	2000
4	1800	9	2050
5	1850	10	2100

The samples were removed at the temperatures indicated and allowed to cool. They were weighed in air and then in mercury, from which data their bulk densities were calculated. By comparing the bulk densities of the samples removed at the various temperatures, the temperature at which the major part of the bloating occurred could be ascertained.

Procedure for Determining the Time of Exposure to a Particular Temperature on the Bloating Characteristics of Shale.- Fifteen samples of the shale designated as "purple" were prepared, each weighing about 25 grams. These samples were thoroughly cleaned, accurately weighed, and numbered from one through fifteen. The temperature of the furnace was raised to 2025° F. over a period of one hour. The furnace was then adjusted so as to maintain this temperature throughout the tests. Samples were removed from the furnace at five minute intervals. Thus, sample #1 was removed when the temperature reached 2025° F.; #2 was removed five minutes later; #3, ten minutes after the temperature reached 2025° F., and so on, until sample #10 was removed forty-five minutes after the

temperature reached 2025° F. The samples were cooled and
weighed and their bulk densities determined by means of the
mercury volumeter. By comparing the bulk densities of the
samples removed from the furnace after various time exposures to the temperature of the furnace, the effect of the
time of exposure to 2025° F. on bloating could be determined.

Procedure for Determining the Effect of Rapid Heating on
the Bloating Characteristics of Shale.- For this test, sample #8-29 was selected since it had already been shown to
bloat well. It was decided to use two furnaces for this test
rather than just one because it was feared that the thermal
shock of injecting the shale into a furnace at 2100° F. would
cause disintegration of and loss of the sample. Accordingly,
two furnaces were used; one, an electric muffle furnace which
was brought to a constant temperature of 1590° F., and the
other, a gas-fired muffle furnace which was brought to a temperature of 2150° F. and held there.

For this test, nine samples of shale #8-29, each weighing about 25 grams, were prepared and numbered 1 through 9.
Samples 4, 5 and 6 were placed in the gas furnace and the
temperature raised to 2150° F. in a period of two hours.
Samples 1, 2 and 3 were placed in the electric furnace which
had previously been brought to a temperature of 1590° F.
The samples were allowed to remain in this furnace at 1590° F.

for ten minutes and were then immediately transferred to the
gas-fired furnace, which was at a temperature of 2150° F.,
and allowed to remain for ten minutes at 2150° F. In this
way, samples 4, 5 and 6 were brought from room temperature to
2150° F. in twenty minutes while samples 1, 2 and 3 were
brought to 2150° F. over a period of two hours. The bulk den-
sities were determined for all six samples and a comparison
made to determine the effect of the quick heating on the
bloating characteristics of the shale. Samples 7, 8 and 9
were used to determine the bulk density of the unburned shale.
This was done with the mercury volumeter.

Chemical Analyses.- Chemical analyses were made on sam-
ples "buff", #8-29 burned and #8-29 unburned. The analyses
included determination of loss on drying at 110° F., loss on
low temperature ignition, loss on high temperature ignition,
silica, combined iron and aluminum oxides, iron oxides, alum-
inum oxide by difference, calcium, magnesium, and combined
alkalies.

The samples selected were chosen because the "buff"
shale exhibited rather poor bloating qualities and shale
#8-29 showed excellent bloating qualities. Thus, by compar-
ing the chemical composition of the two, it was thought that
the compound responsible for the bloating could be deter-
mined.

The samples were prepared for analyses by selecting samples of about 25 grams and grinding them in a mortar until the entire sample would pass the Tyler Standard 200 mesh screen. Standard methods of analyses were used throughout [35].

Comparison of Crushing Strength of Concrete made with Cinder Aggregate and Concrete made with Expanded Shale Aggregate.- For this test, regular production cinders, as used by Cinder Block Inc. of Roanoke, Virginia, were used for making the cinder concrete. Expanded shale, consisting of mixed "buff" and "purple" shale, was used for the expanded shale aggregate. The aggregates were carefully graded into eight size ranges. These ranges and the percentage of total aggregate in each range were: plus 3/8 mesh, 6.34%; minus 3/8 to plus 4 mesh, 40.60%; minus 4 to plus 8 mesh, 15.28%; minus 8 to plus 14 mesh, 9.94%; minus 14 to plus 28 mesh, 7.95%; minus 28 to plus 48 mesh, 4.97%; minus 48 to plus 100 mesh, 6.97%; minus 100 mesh, 7.95%. Tyler Standard screens were used for grading the aggregate.

The ratio of the weight of aggregate to weight of cement was three to one in both instances. The water to cement ratio was eight-tenths to one by weight. Both mixes were mixed in as much the same manner as was possible by hand mixing. The mixed concrete was molded into six inch (diameter) by twelve inch (high) cylinders by filling the mold

half full and tamping 75 times with a ten-pound tamping rod;
then filling to three-fourths full and tamping 100 times;
then filling to three-fourths full and tamping 75 times; then
filling the mold and tamping 75 times; then filling the mold
a second time and tamping 50 times; filling the mold a third
time and tamping 50 times; and finally striking the top off
smooth with a plasterer's trowel.

The cylinders were placed in a curing autoclave in which
the steam pressure was maintained at 150 pounds per sq. in.
gage for a period of eight hours. The cylinders were then
removed from the curing autoclave and were capped with sul-
phur for testing. The cylinders were tested in the compres-
sion testing machine of the Materials Laboratory of Virginia
Polytechnic Institute approximately twenty-four hours after
they were molded.

Data and Results

The data and results of this investigation may be divided into six parts. First, the classification of the shale samples investigated, by rank as to their ability to bloat; second, the investigation of the temperature at which bloating occurs; third, the effect of the time of exposure of a shale sample to 2025° F. on its bloating qualities; fourth, the effect of the rate of heating on the bloating of shale; fifth, the chemical analysis of burned and unburned shale; and sixth, the comparison of the compressive strengths of concrete made with cinders as an aggregate and of concrete made with bloated shale as an aggregate.

The Classification of the Shale Samples Investigated, by Rank as to their Ability to Bloat.- In Table 1 are listed the bulk densities of thirty-two shale samples before being expanded or bloated by heat. These shale samples were obtained from the test holes shown in Fig. 1. All of these samples were exposed to heat as shown in the Firing Schedule, Fig. 5, and were removed from the heat when the temperature reached 2200° F. Table 2 gives the bulk densities of these same samples after they have been exposed to the firing schedule, as shown in Fig. 5. Table 3 gives the bulk densities of the burned and unburned samples listed in Tables 1 and 2 and

Table 1.

Data Used for Calculation of Bulk Densities of Unburned
Shale Samples from Various Test Holes on Ferguson Property in
Roanoke, Virginia.

Sample Number	Weight in Air Gm.	Weight in Water Gm.	Loss in Wt. in Water Gm.	Bulk Density Gm./cc.
1-9	19.6541	12.1475	7.5066	2.618
1-16	33.2620	20.8595	12.4025	2.682
1-26	24.6400	15.3541	9.2859	2.653
1-35	21.9875	13.7500	8.2375	2.669
1-45	33.9407	21.9000	12.0407	2.819
1-55	44.4480	27.8416	16.6064	2.676
1-72	59.5571	37.3744	22.1827	2.685
2-25	14.1525	8.9370	5.2155	2.713
2-31	17.7945	11.1625	6.6320	2.608
2-38	22.4172	13.9503	8.4669	2.647
2-42	9.3814	5.9987	3.3827	2.773
2-50	22.3163	13.6100	8.7063	2.563
3-76	31.9474	20.6410	11.3064	2.825
3-77	25.8296	16.5498	9.2800	2.783
5-5	24.6870	14.6408	10.0462	2.457
5-6	18.0910	11.5461	6.4449	2.805
5-8	25.4686	16.2730	9.1956	2.775
5-10	34.8595	22.2955	12.5640	2.775
5-12	43.5742	28.0885	15.4857	2.815
5-15	21.8704	14.1049	7.7655	2.816
7-18	29.5950	18.7117	10.8833	2.719
7-26	10.6855	6.8440	3.8415	2.781
7-33	22.9165	13.6894	9.2271	2.483
7-41	25.1191	15.7180	9.4011	2.671
7-45	28.3122	17.7326	10.5796	2.676
8-5	51.6950	32.6980	18.9970	2.721
8-12	26.1232	16.6214	9.5018	2.749
8-22	31.3600	20.3765	10.9835	2.855
8-29	30.3130	19.5787	10.7343	2.823
8-42	42.8600	27.6951	15.1649	2.826
Buff	11.5475	7-2148	4.3327	2.665
Purple	7.9260	5.0069	2.9191	2.715

Table 2.

Data Used for Calculation of Bulk Densities of Burned Shale Samples from Various Test Holes on Ferguson Property in Roanoke, Virginia.

Sample Number	Weight in Air Gm.	Weight in Mercury Gm.	Volume of Sample Gm.	Bulk Density Gm./cc.
1-9	28.1535	1325	24.47	1.150
1-16	21.6988	1005	18.59	1.167
1-26	5.5755	280	5.14	1.084
1-35	6.6340	450	8.10	0.819
1-55	4.5472	340	6.08	0.748
1-72	19.4500	1025	18.76	1.037
2-25	27.4225	675	13.43	2.042
2-31	14.7805	580	10.89	1.357
2-38	15.3859	505	9.67	1.591
2-42	12.2886	510	9.53	1.289
2-50	30.2600	1005	19.22	1.574
3-76	8.9742	459	8.42	1.065
3-77	10.0770	452	8.38	1.202
5-5	11.4983	467	8.74	1.315
5-6	4.4608	180	3.37	1.323
5-8	2.2030	100	1.86	1.189
5-10	11.0615	475	8.85	1.252
5-12	12.8810	535	9.99	1.289
5-15	10.6500	543	10.05	1.060
7-18	27.7270	1097	20.59	1.346
7-26	10.3617	428	8.00	1.295
7-33	19.4544	578	11.21	1.736
7-41	11.1985	545	10.04	1.115
7-45	27.6542	734	14.45	1.914
8-5	41.8055	1505	28.52	1.465
8-12	14.4625	521	9.17	1.464
8-22	25.7670	700	13.73	1.876
8-29	17.0377	1103	19.90	0.856
8-42	7.7808	654	11.63	0.669
Buff	35.2736	1029	20.00	1.763
Purple	29.0795	975	18.63	1.5611

Table 3.

Rank of Bloating and Ratios of Bulk Densities of Unburned
to Burned Shale Samples from Ferguson Property in
Roanoke, Virginia.

Rank of Bloating	Sample Number	Bulk Density Unburned Gm./cc.	Bulk Density Burned Gm./cc.	Ratio of Unburned to Burned Bulk Densities
1	8-42	2.826	0.669	4.2241
2	1-55	2.676	0.748	3.5771
3	8-29	2.823	0.856	3.2971
4	1-35	2.669	0.819	3.2581
5	8-15	2.816	1.060	2.656
6	3-76	2.825	1.065	2.652
7	1-72	2.685	1.037	2.589
8	1-26	2.653	1.084	2.447
9	7-41	2.671	1.115	2.395
10	5-8	2.775	1.189	2.333
11	3-77	2.790	1.202	2.321
12	1-16	2.682	1.167	2.298
13	1-9	2.618	1.150	2.276
14	5-10	2.775	1.252	2.215
15	5-12	2.815	1.289	2.183
16	2-42	2.773	1.289	2.151
17	7-26	2.781	1.295	2.147
18	5-6	2.805	1.323	2.120
19	7-18	2.719	1.346	2.020
20	2-31	2.608	1.357	1.921
21	8-12	2.749	1.464	1.877
22	5-5	2.457	1.315	1.868
23	8-5	2.721	1.465	1.857
24	Purple	2.715	1.561	1.739
25	2-38	2.647	1.591	1.663
26	2-50	2.563	1.574	1.628
27	8-22	2.855	1.876	1.522
28	Buff	2.665	1.763	1.512
29	7-33	2.483	1.736	1.430
30	7-45	2.676	1.914	1.398
31	2-25	2.713	2.042	1.328

also the quotient obtained by dividing the unburned bulk density by the burned bulk density. This quotient or ratio of bulk densities provides a method by which the shale samples obtained from the various test holes on the Ferguson Property may be classified as to their ability to bloat when exposed to this particular firing schedule (Fig. 5). Thus, in Table 3, the shale samples are listed in the order of their ability to bloat.

The Investigation of the Temperatures at Which Bloating Occurs.- This investigation was made using three different types of shale; the "buff" shale, #7-33 shale, and the "purple" shale. Separate firings were made for each type of shale but all firings were made according to the firing schedule as shown in Fig. 5. The results of this investigation are shown graphically in Figs. 2, 3 and 4. Thus, in Fig. 2, each point on the curve represents the temperature of the furnace when a sample of the "buff" type of shale was removed from the furnace plotted against the bulk density of the sample. Therefore, the point where the bulk density begins to drop off sharply indicates the temperature at which bloating occurs. Figs. 3 and 4 are plotted in like manner for shales #7-33 and "purple", respectively.
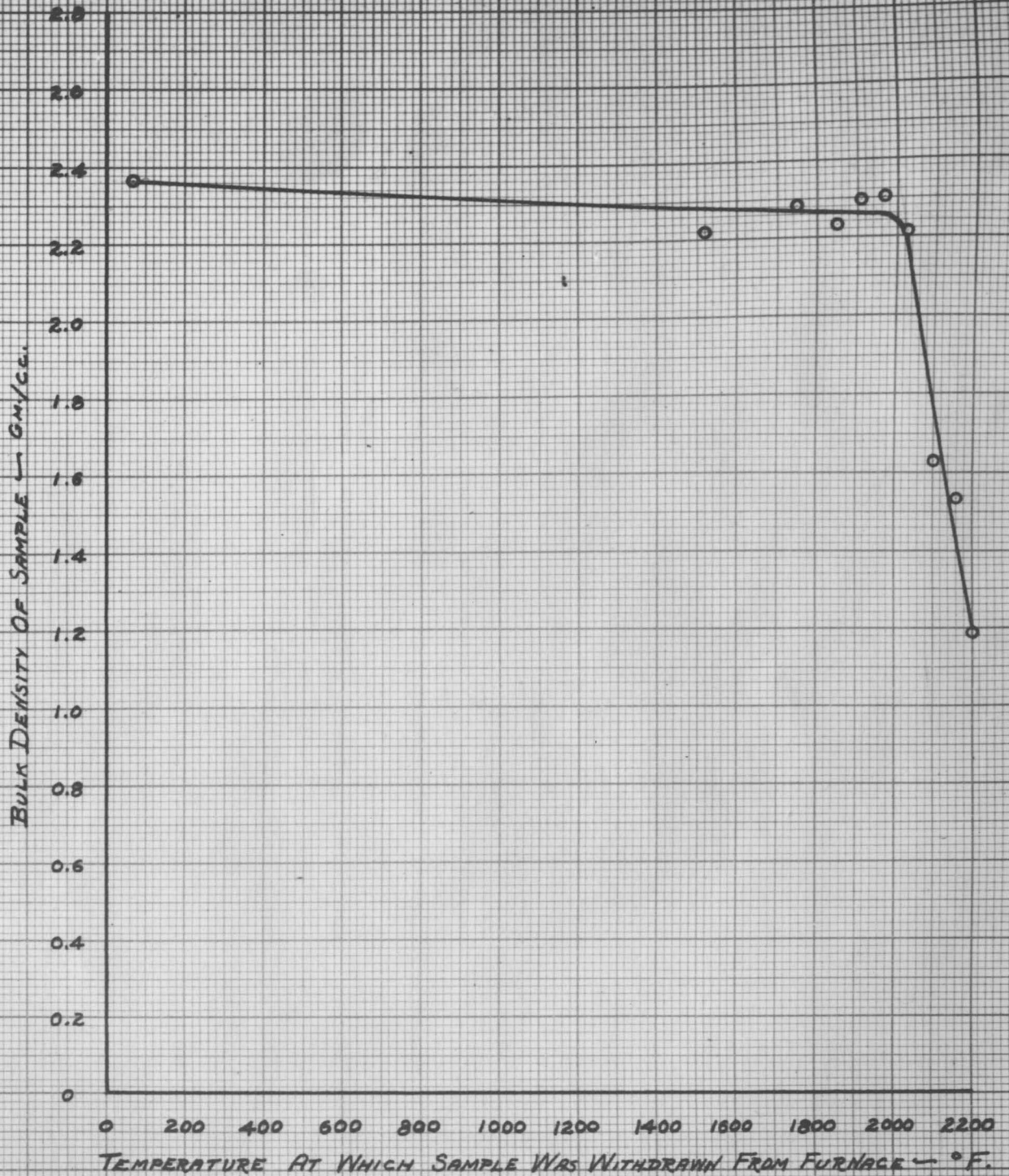
FIG. 2.—RELATIONSHIP OF THE TEMPERATURE AT
WHICH SAMPLE "BUFF" WAS REMOVED FROM THE
FURNACE TO ITS BULK DENSITY.
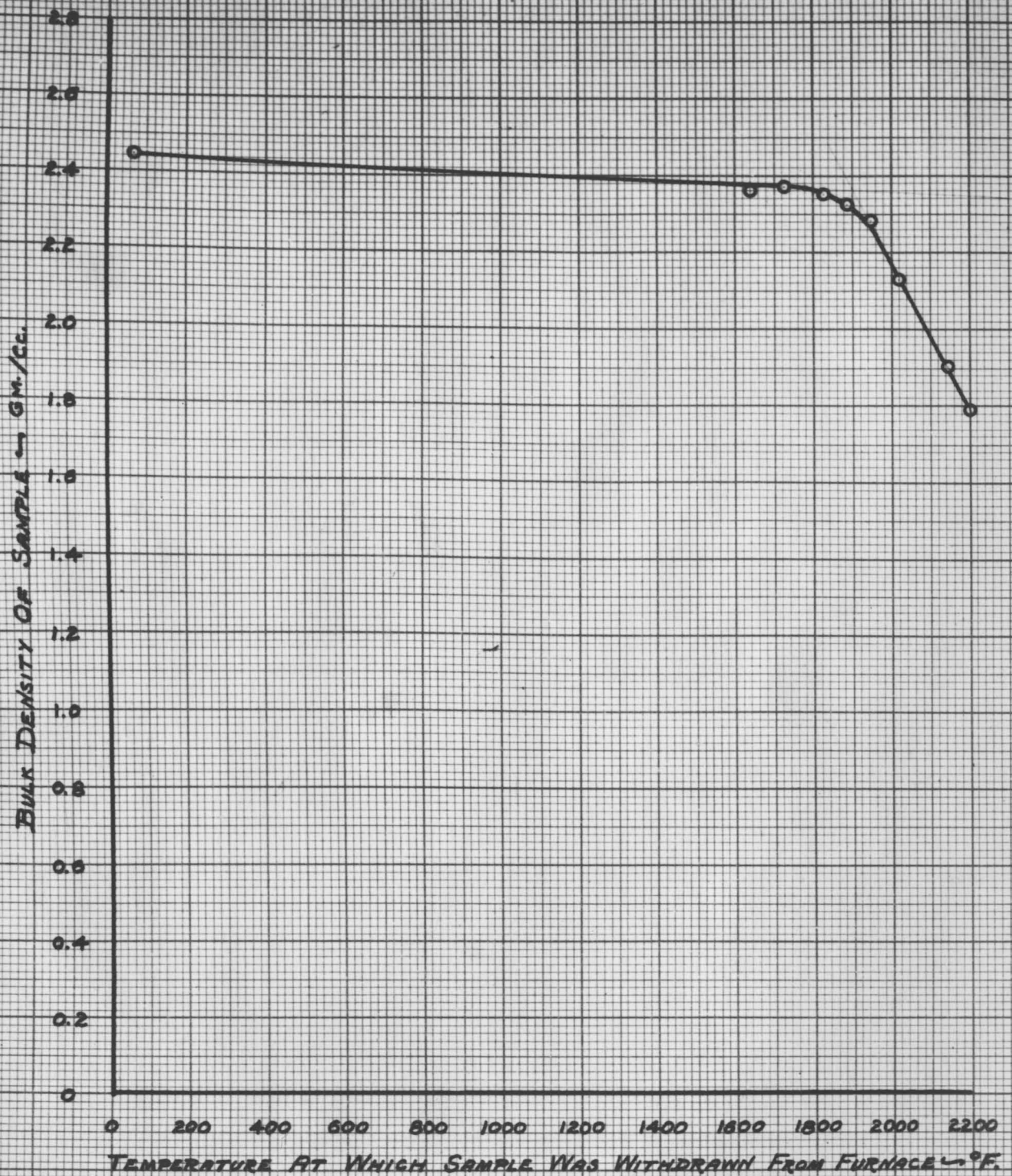
FIG. 3 — RELATIONSHIP OF THE TEMPERATURE AT
WHICH SAMPLE 7-33 WAS REMOVED FROM THE
FURNACE TO ITS BULK DENSITY.
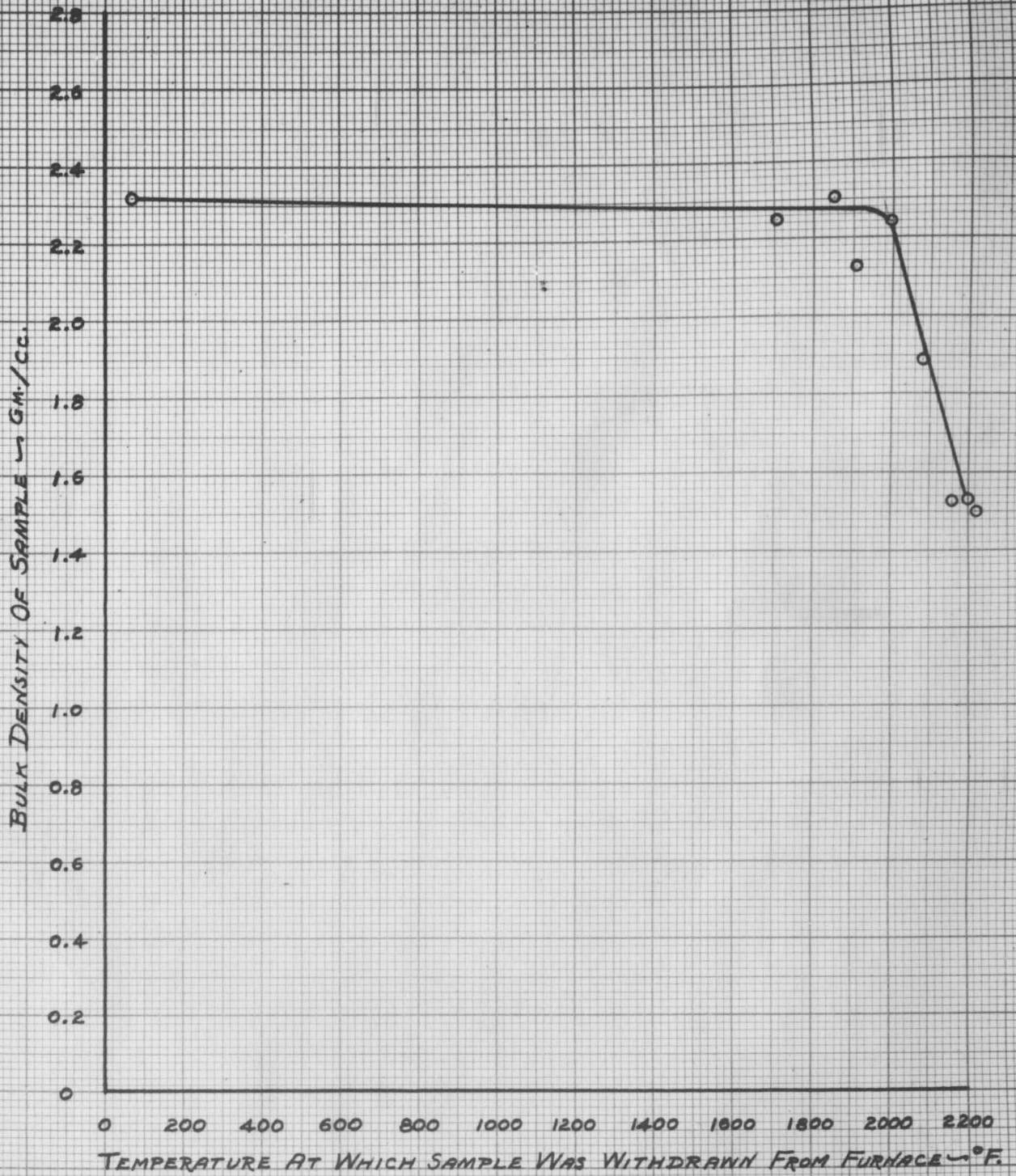
FIG. 4 — RELATIONSHIP OF THE TEMPERATURE AT
WHICH SAMPLE "PURPLE" WAS REMOVED FROM THE
FURNACE TO ITS BULK DENSITY.

The Effect of Time of Exposure of a Shale Sample to
2025° F. on its Bloating Qualities.- The shale designated
as "purple" was used in this test. Fig. 4 shows that the
temperature at which bloating begins for this shale is
1900° F. Therefore, a temperature of 2025° F. was used in
this test to insure that the temperature of bloating was
reached. The temperature of the furnace was increased to
2025° F., as shown in the firing schedule, Fig. 5, but when
this temperature was reached, the furnace was adjusted so as
to maintain a constant temperature of 2025° F. for the dura-
tion of the test.

Samples removed from the furnace after 0, 5, 10, 15, 20,
25, 30, 35, 40, and 45 minutes exposure to 2025° F. had bulk
densities of 1.82, 2.14, 1.89, 1.85, 1.65, 2.19, 1.90, 1.71,
1.83, and 2.08 gm./cc., respectively. The losses in weight
of the samples exposed to 2025° F. for 5, 10, 20, 25, 30,
and 40 minutes were 2.97, 3.41, 3.57, 3.33, 2.91, and 2.99
per cent, respectively.

The Effect of the Rate of Heating on the Bloating of

Shale.- Table 4 shows the bulk densities of shale #8-29 when heated to 2150° F. in a period of 1 hour and 45 minutes, as compared to samples of the same shale when heated to 2150° F. in 20 minutes. This test was made to determine if the rate of heating affected the amount of bloating of shale #8-29.

- 61 -

Table 4.

Bulk Densities of Shale #8-29 when Heated to 2150° F. in
One Hour and Forty-five Minutes Compared to Bulk Densi-
ties when Heated to 2150° F. in Twenty Minutes.

Unburned Shale		Shale whose Temperature was Raised from Room Temperature to 2150° F. in 1 3/4 Hr.		Shale Whose Temperature was Raised from Room Temperature to 2150° F. in Twenty Minutes.	
Sample Number	Bulk Density Gm./cc.	Sample Number	Bulk Density Gm./cc.	Sample Number	Bulk Density Gm./cc.
7	2.725	4	0.581	1	0.469
8	2.685	5	0.639	2	0.629
9	2.745	6	0.648	3	0.474
Average	2.718	Average	0.623	Average	0.524

The Effect of the Chemical Composition of Shale on its
Ability to Bloat.- Table 5 shows the results of chemical
analysis made on the unburned shale sample "buff" which exhib-
its poor bloating characteristics (the ratio of the bulk den-
sities of the unburned "buff" to the burned "buff" is 1.512),
and the chemical analysis of both the burned and unburned
shale #8-29 which exhibits excellent bloating characteristics
(the ratio of the bulk densities of the unburned #8-29 to the
burned is 3.297). This test was made to determine what ele-
ment or compound in the shale is responsible for the bloating
of the shale by comparing the amounts of the various compounds
present in the poor bloating "buff" shale with the amounts of
the various compounds present in the good bloating #8-29
shale.

Comparison of the Compressive Strengths of Concrete made
with Cinders as the Aggregate with Compressive Strengths of
Concrete made with Bloated Shale as the Aggregate.- Table 6
shows the per cent of each size of aggregate used in making
the concrete for the 6-inch diameter by 12-inch high test
cylinders. The concrete mixes were made with the following
proportions by weight: 1 part water to 1.54 parts cement to
4.38 parts aggregate. The test cylinders, three of cinder
concrete and three of bloated shale concrete, were cured in
steam at a pressure of 150 lbs./sq.in. gage for eight hours

Table 5.

Chemical Analyses of Shale Samples "Buff" and #8-29 from
Ferguson Property, Roanoke, Va.

	Buff	#8-29 Burned	#8-29 Unburned
Loss on drying at 212° F., %	0.22	0.00	0.69
Loss on heating at 800-900° F., %	0.22	0.00	1.00
Loss on heating at 1700-1800° F., %	0.76	0.00	1.51
Silica, %	63.24	53.57	49.60
Ferric Oxide, %	2.33	4.62	3.75
Aluminum Oxide, %	32.75	29.45	29.16
Calcium Oxide, %		4.28	
Magnesium Oxide, %		3.72	
Calcium Carbonate, %	Trace		5.73
Magnesium Carbonate, %	Trace		7.51
Undetermined	0.48	4.36	1.05
Total	100.00	100.00	100.00

Table 6.

Grading of Aggregate Used to Make Concrete for Compressive
Strength Test Cylinders - Used to Compare Strengths of
Cinder Concrete and Bloated Shale Concrete.

Tyler Standard Screen No.		Differential Amount Retained on Screen %	Cumulative Amount Retained on Screen %
Passing	Retained on		
	3/8	6.34	6.34
3/8	4	40.60	46.94
4	8	15.28	62.22
8	14	9.94	72.16
14	28	7.95	80.11
28	48	4.97	85.08
48	100	6.97	92.05
100	Pan	7.95	100.00

Table 7.

Compressive Strengths of Concrete Test Cylinders Made with
Cinders as an Aggregate and of Concrete Test Cylinders
Made with Bloated Shale from the Ferguson Property
in Roanoke, Virginia, as an Aggregate.

Cinder Concrete				
Sample Number	Weight Lb.	Cinder Concrete Density Lb./cu.ft.	Ultimate Load - Lb.	Unit Strength Lb./sq.in.
C1	18.38	93.60	58,000	2051
C2	18.33	93.35	60,000	2122
C3	18.35	93.45	54,000	1910
Average	18.33	93.47	57,333	2028
Bloated Shale Concrete				
Sample Number	Weight Lb.	Bloated Shale Concrete Density Lb./cu.ft.	Ultimate Load - Lb.	Unit Strength Lb./sq.in.
S1	22.39	114.03	117,000	4138
S2	22.59	115.04	121,000	4279
S3	22.35	113.82	123,000	4350
Average	22.44	114.30	120,333	4256

and were tested in compression at an age of twenty-four
hours.   The results of the compressive strength tests, as
well as the weights and bulk densities of the concrete are
shown in Table 7.   These tests were designed to provide a
comparison betwen cinder concrete and bloated shale concrete
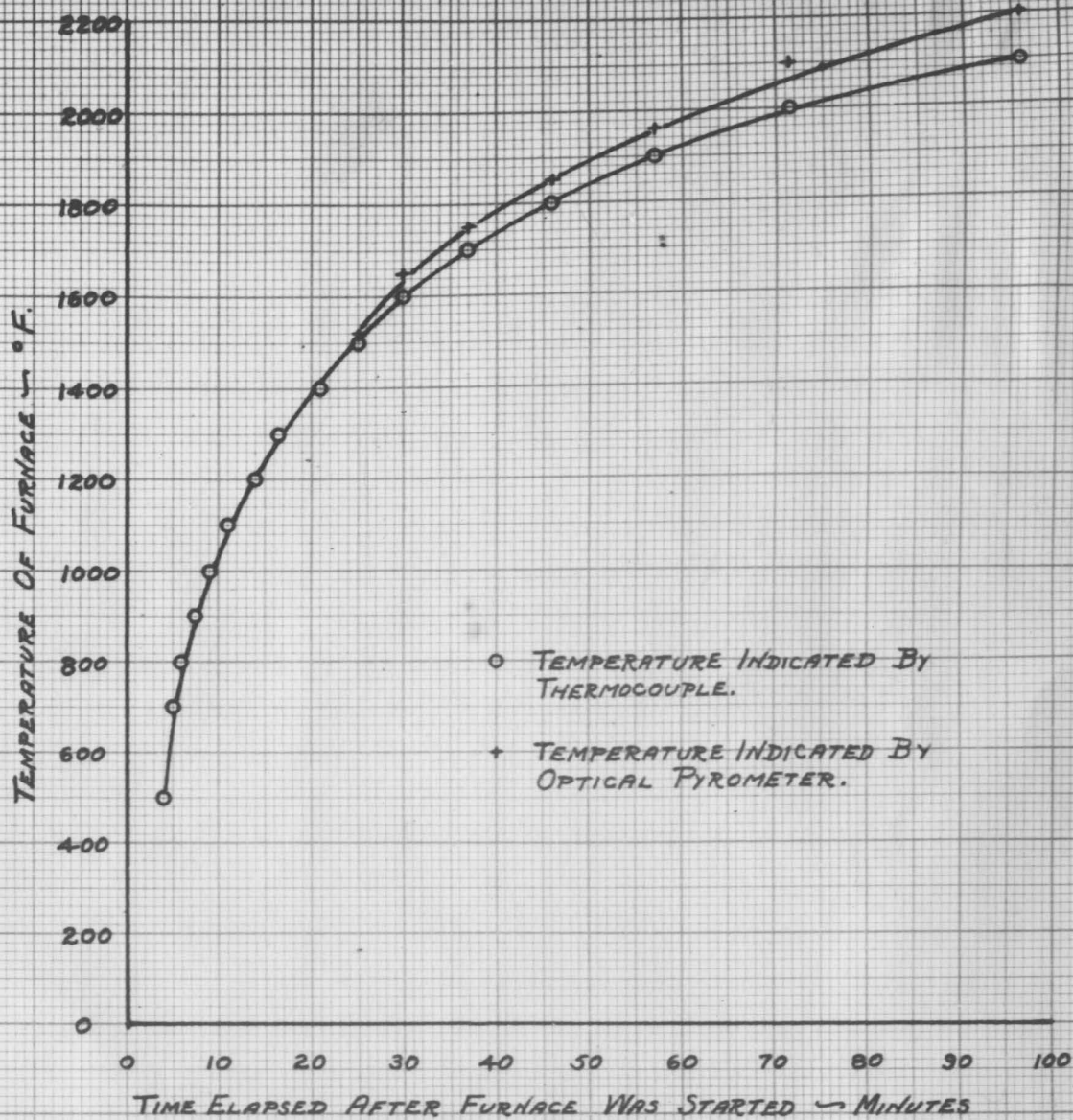as far as compressive strength is concerned.

FIG. 5 — FIRING SCHEDULE — THE AVERAGE RATE OF TEMPERATURE INCREASE USED TO BLOAT THE SHALE LISTED IN TABLES 1, 2, AND 3 AND FIGURES 2, 3, 4, 5, AND 6.
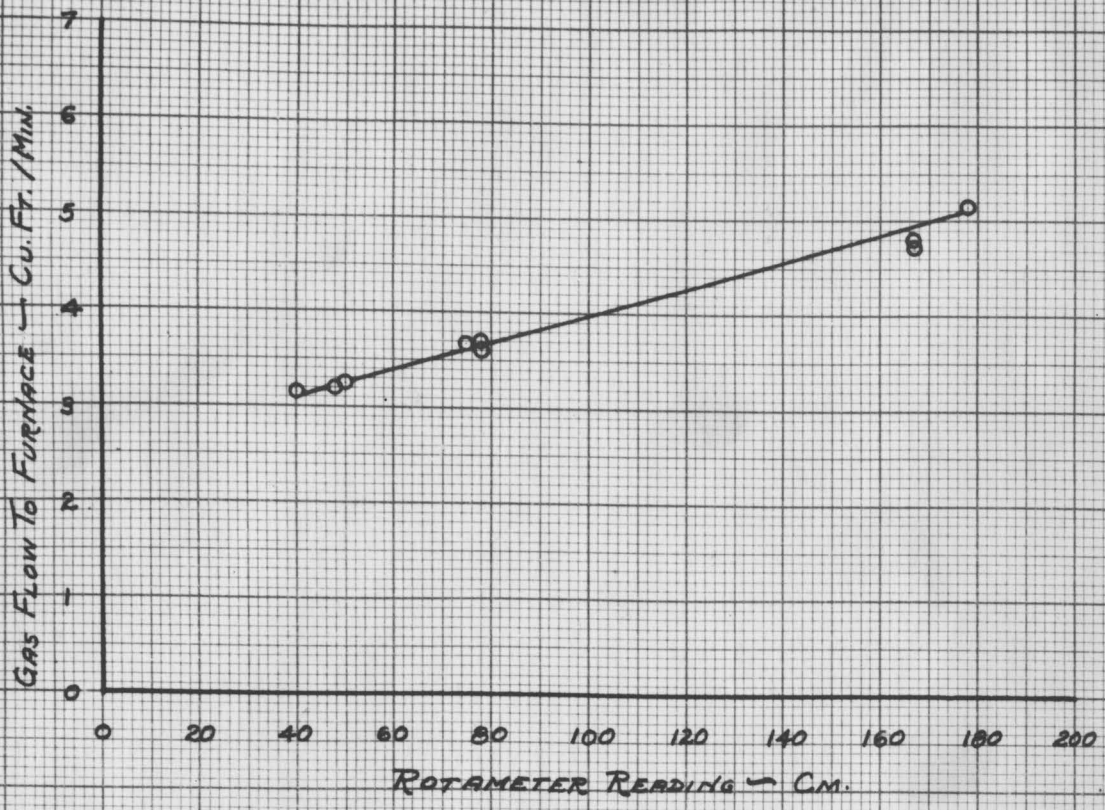
FIG. 6 — CALIBRATION CURVE OF ROTAMETER USED TO
MEASURE THE GAS FLOW TO THE MUFFLE FURNACE. CALIBRATION
WAS MADE WITH A SARGENT WET TEST GAS METER.

IV. DISCUSSION OF RESULTS

Ferguson Property Shale as a Source of Lightweight Aggregate.- Table 3 lists the shale samples from the various test holes in the order of their ability to bloat. As an index to the bloating ability of each sample, the ratio of the bulk densities of the unburned sample to the burned sample was used. This ratio indicates the number of times lighter the sample became when exposed to the firing schedule, as shown in Fig. 5.

Test Hole #1.- From the results shown in Table 3, it is seen that no clear-cut relation can be obtained between the degree to which a shale will bloat and its location on the Ferguson Property. Neither can such a relation between the degree to which a shale will bloat and its depth in the ground be determined. However, it is to be noted that of the thirty-one samples tested, no sample from test hole #1 was below thirteenth in rank in ability to bloat. The best bloating shale from test hole #1 was found at a depth of 55 feet. The ratio of the bulk density of the unburned shale #1-55 to the bulk density of the burned shale #1-55 was 3.577. The poorest bloating shale from test hole #1 was #1-9 which had a ratio of bulk densities of 2.276. It is interesting to note

that this sample was obtained nearest to the surface of any
of the shale samples from test hole #1. This suggests that
the degree of bloating will increase as the depth from which
the sample is taken is increased. This idea is further borne
out by the fact that sample #1-16 and #1-26, which were the
next two samples taken after #1-9, are immediately above #1-9
in rank of bloating. However, the shale just above #1-26 in
bloating ability was #1-72 which was obtained at the bottom
of test hole #1. The next two samples above #1-72 in bloat-
ing ability are #1-35 and #1-55, respectively. The last three
samples from test hole #1 tend to discount the theory that as
the depth from which the sample is obtained is increased, the
bloating ability will increase.

A sample taken at a depth of 45 feet from test hole #1
failed to bloat and crumbled after being fired according to
the firing schedule in Fig. 5. This indicates that a seam of
shale at 45 feet should be avoided should the shale from this
area be used commercially as a source of lightweight aggre-
gate. The maximum thickness of this strata of poor shale is
20 feet since the samples taken from 35 feet and 55 feet
showed excellent bloating characteristics. Otherwise, the
shale from test hole #1 may be made to bloat to a bulk densi-
ty varying from 0.748 gm./cc at 55 feet to a bulk density of
1.167 gm./cc. at 16 feet by exposing it to a firing schedule

as shown in Fig. 5. A bulk density of approximately 1 gm./cc. is desirable for a lightweight aggregate since this lessens the tendency for the aggregate to sink to the bottom or rise to the top of the wet concrete mass. Also, the range of the bulk densities of the shale from this test hole is less than that of cinders thus indicating that a more uniform aggregate may be obtained. The bulk density of cinders as used for cinder concrete varies considerably. Part of the so-called cinders consists of a very light coke-like material which has a bulk density of about 0.5 gm./cc. On the other hand, glass-like slag is often found in the cinders which has a bulk density of more than 2 gm./cc. Of course, other material may be found in the cinders having bulk densities of anywhere between these extremes of 0.5 gm./cc. to 2 gm./cc. The average bulk density of cinders is approximately 1.4 gm./cc.

The appearance of the burned shale from test hole #1 was all very dark in color. The apparent strength of the burned shale was good, it being impossible to crush or break particles by hand, including samples #1-55 and #1-35, which bloated to bulk densities of 0.748 and 0.819, respectively. The texture of the burned shale was uniform, having pore sizes of approximately 1/16-inch diameter for shales #1-55 and #1-35 to approximately 1/32-inch for the shale #1-9 and #1-16. Also, it was noted when the samples were examined under a mic-

roscope that the pores or cells of the bloated shale were not interconnecting. This was further borne out by placing samples of burned shale #1-55 and #1-35 in water (bulk densities 0.748 gm./cc. and 0.819 gm./cc., respectively). These samples floated on the water for a period of one month and would still float when they were removed at the end of a month.

Test Hole #2.- The shale samples from test hole #2 did not show good bloating characteristics. This was due to the high limestone and other carbonate content of these shales. Although a quantitative test for limestone was not made on these samples, the fact that effervescence occurred when the samples were treated with acid, together with the fact that burning of the samples, according to the firing schedule in Fig. 5, caused the samples to become powdery and white, was considered ample evidence that the samples were too high in limestone or other carbonates to be of use as a lightweight aggregate. The general appearance of these samples after being burned was a cellular structure in which this powdery white substance was deposited. Only one sample from test hole #2 disintegrated so badly that determination of the bulk density was impossible. This was sample #2-20. The best bloating sample from test hole #2 was #2-42, which ranked sixteenth in the thirty-one samples tested and had a ratio of unburned bulk density to burned bulk density of 2.151. Sample #2-31

ranked twentieth and had a bulk density ratio of 1.921. Samples #2-38 and #2-50 ranked twenty-fifth and twenty-sixth, respectively, and had bulk density ratios of 1.591 and 1.574. Sample #2-25 was the poorest bloating shale of the thirty-one samples tested, the burned shale having a bulk density of 2.042 and a ratio of unburned bulk density to burned bulk density of only 1.328. In view of the results of tests on the shale from test hole #2, it is not deemed advisable to use the shale from this area for the production of light-weight aggregate.

Test Hole #3.- Very little shale was encountered in test hole #3 and this was at a depth of 75 feet. Drilling was discontinued at 77 feet. Only two samples were tested from this location, one being taken from 76 feet and the other from 77 feet. Sample #3-76 burned to a bulk density of 1.065 and the ratio of unburned to burned bulk densities was 2.652. This sample ranked sixth in the thirty-one samples tested in ability to bloat. Sample #3-77 ranked eleventh and burned to a bulk density of 1.202 with a ratio of bulk densities of 2.321. Thus, the shale that was found at this extreme depth was of good quality as far as bloating is concerned. The pores of the bloated product were of uniform size (approximately 1/32-inch diameter) and the apparent strength was good. It was not possible to crush or break the samples

by hand.

Test Hole #4.- Test hole #4 did not yield a shale that could be sampled by core drilling and no tests were made on shale from this hole.

Test Hole #5.- Test hole #5 yielded shale of varying quality. Sample #5-15 bloated well, ranking fifth in the thirty-one samples tested and having a ratio of unburned to burned bulk density of 2.685. Sample #5-8 ranked tenth with a ratio of 2.333. Samples #5-10 and #5-12 ranked fourteenth and fifteenth with ratios of 2.22 and 2.199, respectively. Sample #5-6 ranked eighteenth with a ratio of 2.12 and sample #5-5 ranked twenty-second with a ratio of 1.87. The range of bulk densities of the samples tested from test hole #5 was from 1.060 gm./cc. for sample #5-15 to 1.315 for sample #5-5. This range of bulk densities is much more narrow than the range of bulk densities in cinders that are ordinarily used for concrete aggregate. Therefore, the lightweight aggregate made from shale from this location would provide a more uniform product than cinders.

Test Hole #6.- Test hole #6 yielded only a stiff red clay that burned without bloating to a brick-like product. Bulk density tests were not made on this product.

Test Hole #7.- Test hole #7 yielded a shale that did not bloat very well. The best bloating sample taken from this hole was from a depth of 41 feet. Sample #7-41 ranked ninth in the thirty-one samples tested and the ratio of its unburned bulk density to its burned bulk density was 2.395. Sample #7-26 ranked seventeenth with a ratio of 2.147. Sample #7-18 ranked nineteenth with a ratio of 2.02. Samples #7-35 and #7-45 ranked twenty-ninth and thirtieth with ratios of 1.398 and 1.328, respectively. The range of bulk densities was from 1.115 to 1.914. Since shale was encountered the entire depth of this hole (45 feet) there is evidently a large amount of shale present in this location. The bulk densities of the burned shale is high but it is believed that this shale could be successfully used as a source of lightweight aggregate.

Test Hole #8.- Test hole #8 produced a shale of excellent bloating quality from a depth of 29 feet through a depth of 42 feet. Sample #8-42 ranked first among the thirty-one samples tested and the ratio of the unburned bulk density to the burned bulk density was 4.224. Sample #8-29 ranked third with a ratio of 3.297. The appearance of these two bloated shales was the same. The color was a glossy medium gray and the pore size relatively large (about 1/8-inch diameter). The strength of these bloated shales was not as good as #1-55

or #1-35 as it was possible to break the samples and crush
them in the hand. However, the pores were not interconnect-
ing and it is believed that a non-load-bearing concrete of
very light weight could be produced from this bloated shale.
Samples from test hole #8 from 5 feet to 22 feet did not
bloat very well. Sample #8-12 ranked twenty-first with a ra-
tio of 1.877. Sample #8-5 ranked twenty-third with a ratio
of 1.857. Sample #8-22 ranked twenty-seventh with a ratio
of 1.529. These latter three samples from test hole #8, while
not producing as light a product as desired (the bulk densi-
ties ranked from 1.464 to 1.876), were of high strength and
uniform texture. It is believed that a very strong concrete.
could be made with this aggregate but one which would be rel-
atively heavy for a lightweight concrete.

The samples "buff" and "purple", which were obtained from
the face of a road cut through the Ferguson Property, did not
exhibit good bloating. Sample "purple" ranked twenty-fourth
with a ratio of unburned to burned bulk density of 1.739.
Sample "buff" ranked twenty-eighth with a ratio of 1.512.
The bloated products from both of these shales were similar
in appearance, being glossy black in color and having pores
less than 1/32-inch in diameter. The strength was very good
as judged by an attempt to break samples with the hand.

<u>Determination of the Temperature of Bloating.</u>- In this
test it was desired to determine the temperature at which the
poorer bloating shales expanded since any piece of apparatus
used to bloat the shales from the Ferguson Property would
have to be operated at the temperature required to bloat the
poorest shale. Therefore, samples "buff", #7-33, and "pur-
ple" were selected for these tests. Fig. 2 shows graphically
the results of a test in which nine samples of the "buff"
shale were burned according to the firing schedule as shown
in Fig. 5. One sample of the "buff" shale was withdrawn from
the furnace as the temperature reached each of the following
points: 1520° F., 1750° F., 1850° F., 1910° F., 1970° F.,
2015° F., 2100° F., 2160° F. and 2200° F. The bulk density
of each of the samples was determined and plotted against the
temperature at which it was withdrawn from the furnace, as
shown in Fig. 3. The resulting curve shows that from room
temperature to a temperature of 2015° F., the bulk density
decreased only from 2.37 gm./cc. to 2.22 gm./cc. while from a
temperature of 2015° F. to 2200° F. the bulk density de-
creased from 2.22 gm./cc. to 1.19 gm./cc. Thus, it is seen
that very little bloating occurred before the temperature
reached 2015° F. and practically all of the bloating occurred
in the temperature range from 2015° F. to 2200° F.

The test made on shale #7-33 (Fig.3) was similar to the

test made on the "buff" shale. In this case, the bulk density decreased from 2.45 gm./cc. at room temperature to 2.3 gm./cc. at 1950° F., while it decreased from 2.3 gm./cc. to 1.79 gm./cc. in the temperature range from 1950° F. to 2200° F.

The "purple" shale (Fig. 4) decreased in bulk density from 2.32 gm./cc. to 2.24 gm./cc. from room temperature to 2000° F. and then decreased from 2.24 gm./cc. to 1.5 gm./cc. in the range from 2000° F. to 2200° F.

Thus, it is seen that none of these samples bloated appreciably until a temperature above 1950° F. was reached. Each curve (Figs 2, 3 and 4) broke sharply downward when this temperature was reached, indicating that the minimum temperature for bloating of the shales tested is 1950° F. when heated according to the firing schedule shown in Fig. 5.

Determination of the Effect of Time of Exposure to 2025° F. on the Bloating of Shale Sample "Purple".- As shown in the preceding section, bloating began with the "purple" shale at 2000° F. (Fig. 4). It was desired to determine if prolonged exposure to this temperature would increase the bloating of the shale. Accordingly, ten samples of "purple" shale were placed in the furnace and the temperature raised according to the firing schedule (Fig. 5) to 2025° F. The temperature was maintained at this point for the duration of

the test. Samples were removed from the furnace after being exposed to 2025° F. for 5, 10, 15, 20, 25, 30, 35, 40, and 45 minutes. The bulk density of each sample was determined to indicate the extent of bloating.

This test was made with the expectation that the longer a particular sample was exposed to this temperature of 2025° F., the greater the degree of bloating that could be expected. However, it was also conceived that after exposure for a certain period of time, bloating would cease and the bulk density of the sample would begin to increase due to fusing of the sample. Contrary to expectations, however, no definite relation could be found between the degree of bloating of the shale sample "purple" and the time of exposure to 2025° F. This is brought out by the results of the tests which show that the bulk density of the shale when exposed to 2025° F. for 5 minutes was 2.14 gm./cc. For a sample exposed to 2025° F. for 10 minutes, the bulk density was 1.89 gm./cc.; 15 minutes, 1.85 gm./cc.; 20 minutes, 1.65 gm./cc.; 25 minutes, 2.19 gm./cc.; 30 minutes, 1.90 gm./cc.; 35 minutes, 1.71 gm./cc.; 40 minutes, 1.83 gm./cc.; and 45 minutes, 2.08 gm./cc.

From these results, it is seen that no regular increase or decrease in bulk density can be expected by reason of heating the "purple" shale at 2025° F. for periods up to 45 min-

utes.

Determination of the Effect of Rate of Heating on the
Bloating of Shale #8-29.- This test was made to determine
the effect of heating the shale rapidly to 2150° F. on its
bloating. The temperature of three samples of shale #8-29
was raised from room temperature to 2150° F. in a period of
20 minutes, after which time the samples were removed from
the furnace. The temperatures of three other samples of
shale #8-29 were raised to 2150° F. over a period of one
hour and 45 minutes according to the firing schedule in Fig.
5. Table 4 gives the results of these tests and it is seen
that the shale bloated to a bulk density of 0.524 gm./cc.
when the temperature was raised to 2150° F. in 20 minutes
while it bloated to a bulk density of 0.623 gm./cc. when the
temperature was raised to 2150° F. over a period of one hour
and 45 minutes. Therefore, the rapid heating does not de-
crease the amount of bloating but, in this case, the rapidly
heated samples were lower in bulk density than the slowly
heated samples. It is possible that this was due to a varia-
tion in the samples but since all of the samples were taken
from the core drilling of test hole #8 and were very close
together, this seems unlikely. It is believed that the slow-
ly heated samples bloated to a maximum point and then, due

to continued heating, began to fuse and run together and in this way experienced this slight increase in density. On the other hand, the rapidly heated shales were not exposed to 2150° F. for a long enough period to undergo this fusion and resulting increase in bulk density.

Effect of Chemical Composition on the Bloating Qualities of Shale.- Chemical analyses were made of two of the shale samples. The "buff" shale was selected as typical of a poor bloating shale and shale #8-29 was selected as typical of a good bloating shale. It was reasoned that, by comparing the chemical analyses of these two shales, the compound or compounds responsible for the superior bloating of shale #8-29 could be detected. Table 3 shows the results of the chemical analyses. Heating of the samples at 800°-900° F. is designed to drive off some water of hydration, decompose magnesium carbonate to the oxide, and decompose organic matter. Any of these actions would liberate gas which might be suspected of causing the bloating of the shale. However, since it has been shown (Figs. 2, 3 and 4) that bloating does not occur until a temperature in excess of 1950° F. is reached, it is not believed that the gases evolved at 800°-900° F. are responsible for the bloating. Therefore, the fact that shale #8-29 contained 7.5% magnesium calculated as the carbonate and lost 1.51% on high temperature ignition while the "buff" shale con-

tained only a trace of magnesium and lost only 0.76% on high temperature ignition, is not believed to have caused the difference in bloating of the two shales. Shale #8-29 contained only 49.6% silica while the poorest bloating "buff" shale contained 63.24% silica. The manner in which this difference in silica content could account for the difference in bloating is not evident. The iron content as the oxide of #8-29 shale was 3.75% and 2.33% for the "buff" shale. The aluminum content as the oxide of the #8-29 shale was 32.75% and 29.16% for the "buff" shale. These differences were so small that it is not believed that the difference in bloating is due to iron or aluminum content. From the determinations made, no logical explanation can be made of the cause of bloating.

Comparative Compressive Strengths of Concrete made with Cinder as an Aggregate and of Concrete made with Bloated Shale as an Aggregate.- Table 6 gives the results of compressive strength tests made on cinder concrete cylinders and bloated shale concrete cylinders. As shown in the table, the weights of the individual cylinders made with the cinder concrete did not vary more than 0.05 lb. while the cylinders made with the bloated shale varied less than 1/4 lb. Thus, it is believed that the method of making and tamping the concrete cylinders was quite adequate for this test. The bulk density of the cinder concrete was 93.47 lb./cu.ft. while the bulk density

of the bloated shale concrete was 114.3 lb./cu.ft. The bloat-
ed shale concrete was, therefore, 20.83 lb./cu.ft. heavier
than the cinder concrete. Ordinary sand and gravel concrete
weighs approximately 145 lb./cu.ft., which is more than 30
lb./cu.ft. heavier than the bloated shale concrete tested.
The reason for the bloated shale concrete being heavier than
the cinder concrete was due to the fact that in preparing the
bloated shale for the concrete, it was necessary to place a
large quantity of the shale in the furnace in order to bloat
enough for the test cylinders. The result was that some of
the shale failed to bloat, particularly that on the inside of
the pile in the furnace.

The average compressive strength of the three cinder con-
crete cylinders was 2028 lb./sq.in., while the average com-
pressive strength of the three bloated shale cylinders tested
was 4256 lb./sq.in. The bloated shale concrete was more than
twice as strong as the cinder concrete but some of this differ-
ence in strength is due to the fact that all of the aggregate
in the bloated shale concrete did not expand thus making the
concrete more dense and stronger than if all of the aggregate
had been properly bloated. In spite of this, the compressive
strengths obtained are amply evidence that a superior concrete
can be made from a mixture of the bloated "buff" and "purple"

shales to that which can be made from commercial cinders now available for making concrete.

Recommendations

The following recommendations are made for future investigation of bloated shale as a lightweight concrete aggregate:

1. That a rotary kiln capable of handling between 30 and 50 lbs. of shale per hour be designed and constructed to bloat the shale. Also, the lining of the kiln should be of a material capable of withstanding temperatures to 2500° F.

2. That the shale samples be subjected to temperatures above 2200° F. to determine the temperature at which bloating ceases and the bulk density begins to increase again.

3. That a thorough chemical analysis be made of a good bloating shale and a poor bloating shale, which includes the determination of the alkali metals, to determine the effect of their fluxing action on the bloating of the shale.

4. That enough shale of each type be bloated in order to make concrete test cylinders so that a comparison can be made of the strengths of concrete produced from aggregates of different bulk densities.

5. That tests be made to determine if the type of atmosphere (oxidizing or reducing) affects the bloating shale.

Limitations

The size of the muffle furnace available for this investigation limited the number of shale samples which could be bloated at one time to fifteen or twenty 25-gram samples. Three concrete test cylinders, six inches in diameter by twelve inches high, require approximately fifty pounds of aggregate. Therefore, it was found impractical to attempt to bloat enough of each of the various shales to make concrete test cylinders so that comparative strength tests could be made. It was found, when the muffle furnace was loaded with approximately twenty pounds of shale crushed to a size that would pass a #3/8 Tyler Standard screen, that much of the shale on the interior of the pile did not bloat. The bloated shale aggregate for the three concrete test cylinders that were made required much hand picking to remove the unbloated shale pieces. It is for this reason that future investigators in this field are advised to employ a rotary kiln capable of handling between thirty and fifty pounds of shale per hour.

Figs. 2, 3 and 4 show that bloating continues with the

shales tested until the maximum temperature (2200° F.) used
in this investigation was reached. It would be desirable to
know the temperature at which this decrease in bulk density
ceases and the temperature at which the bulk density begins
to increase again due to fusion of the shale. The lining of
the muffle furnace used would not withstand temperatures in
excess of 2200° F. One attempt to raise the temperature to
2300° F. resulted in severe damage to the lining and caused
plans for further tests above 2200° F. to be abandoned.

The narrow limits of adjustment of the air to gas ratio
of the muffle furnace made investigation of the effects of
an oxidizing or reducing atmosphere on the bloating of shale
very difficult. It was found that it was impossible to at-
tain temperatures above 2000° F. with the furnace with either
a very small excess or a very small deficiency of air. The
air to gas ratio had to be adjusted so as to produce a flame
with a light blue inner cone and a darker blue outer cone or
else the furnace would not attain temperatures in excess of
2000° F.

As seen in the firing schedule (Fig. 5), the temperature
increase from room temperature to 1500° F. was very rapid,
requiring only 25 minutes. This rapid rise in temperature
was the minimum rate at which the furnace could be operated
as the needle valve gas control on the furnace was only

slightly opened in this range. This condition caused some of the shale samples to disintegrate due to the formation of steam in their interiors. All samples were weighed before being place in the furnace and weighed again after being bloated in order that the loss in weight due to evolved gases could be determined. However, due to the rapid heating up to 1500° F. with the resulting disintegration of many of the samples, this information was not obtained.

V. CONCLUSIONS

From the results of the investigation of the bloating qualities of thirty-one shale samples taken from the Ferguson Property (Fig.1) in Roanoke, Virginia, the following conclusions can be drawn:

1. For the best bloating shale tested, the bulk density was reduced from 2.826 gm./cc. to 0.669 gm./cc. and for the poorest bloating shale the bulk density was reduced from 2.713 gm./cc. to 2.042 gm./cc. when heated in a furnace to 2200° F. over a period of one hour and 36 minutes. The average reduction in bulk density for the thirty-one shale samples tested was from 2.711 gm./cc. to 1.310 gm./cc.

2. The average reduction in bulk density of the shale samples "buff", #7-33 and "purple" when brought to a temperature of 2200° F. in a period of one hour and 36 minutes is from 2.38 gm./cc. to 1.50 gm./cc. Of this reduction in bulk density, only 12.5% occurred when the samples were brought from room temperature to 1900° F., while 87.5% of the reduction in bulk density occurred while the temperatures of the samples were being raised from 1900° F. to 2200° F.

3. Shale sample "purple" does not decrease in bulk density by reason of prolonged heating up to 45 minutes at a

temperature of 2025° F. The bulk density of a sample of the "purple" type shale when heated only five minutes at 2025° F. was 1.89 gm./cc., while another sample heated for 30 minutes at 2025° F. was 1.90 gm./cc.

4. The bulk density of shale #S-29 when heated to a temperature of 2150° F. in a period of one hour and 45 minutes is greater (0.623 gm./cc.) than when heated to 2150° F. in a period of 20 minutes (0.524 gm./cc.)

5. Cinder concrete made from commercial power plant cinders obtained from Cinder Block Inc. of Roanoke weighed 93.4 lb./cu.ft. and had a compressive strength of 2028 lb./sq.in., while concrete made from a mixture of "purple" and "buff" bloated shale weighed 114.3 lb./cu.ft. and had a compressive strength of 4256 lb./sq.in. Both concrete mixes were made in the proportions of 1 part water to 1.54 parts cement to 4.38 parts aggregate by weight. Both the cinder aggregate and the bloated shale aggregate were separated into eight size ranges in identical amounts. Both types of concrete were made the same day, cured in the same manner and tested in compression on the same day.

VI. SUMMARY

This investigation was undertaken to determine the feasibility of producing a cellular, lightweight product by subjecting clay shales to temperatures in the region of 2200° F. Such a product can be utilized as an aggregate for lightweight concrete to supplant cinders which have long been used. The growing scarcity of cinders and the inferior quality of the cinders now obtainable account for the demand for another material to replace them.

A muffle furnace capable of withstanding temperatures to 2200° F. was used to heat the shale samples (25 grams each) which were obtained from the Ferguson Property in Roanoke, Virginia. The degree of bloating or expansion of the shales was measured by determining the bulk density of the shale both before and after exposure to 2200° F.

The best bloating shale investigated bloated from a bulk density of 2.826 gm./cc. to 0.669 gm./cc., while the poorest bloating shale bloated from 2.713 gm./cc. to 2.042 gm./cc. It was also found by a test on three types of shale that of the total bloating occurring when the temperature of the shale is raised to 2200° F., only 12.5% occurs in the temper-

ature range from room temperature to 1900° F., while 87.5%
occurs in the range from 1900° F. to 2200° F. When the tem-
perature of one particular shale was raised to 2025° F. over
a period of one hour and then held at 2025° F. for 45 min-
utes, no additional bloating was noted by reason of maintain-
ing the temperature for 45 minutes. The shale had the same
bulk density at the beginning of the 45-minute period that
it did at the end. A shale sample heated to 2150° F. in 20
minutes bloated to a bulk density of 0.524 gm./cc., while
another sample of the same type heated to 2150° F. in a peri-
od of one hour and 45 minutes bloated to a bulk density of
0.623 gm./cc. Concrete test cylinders made from cinder con-
crete and bloated shale concrete and tested in compression
showed strengths of 2028 lb./sq.in for the cinder concrete
and 4256 lb./sq.in. for the bloated shale concrete. The cin-
der concrete weighed 93.47 lb./cu.ft. while the bloated
shale concrete weighed 114.3 lb./cu.ft. Ordinary sand and
gravel concrete weighs approximately 145 lb./cu.ft.

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