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The Properties of Feldspars and Their Use in Whitewares

by

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Ralph L. Cook
A REPORT OF AN INVESTIGATION

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THE ENGINEERING EXPERIMENT STATION
UNIVERSITY OF ILLINOIS

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The Properties of Feldspars and Their Use in Whitewares

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I. INTRODUCTION

1. Definition
Feldspars are the most common constituents in crystalline rocks and make up about 60 percent of the earth's crust. They may be technically defined as aluminosilicates of sodium, potassium, calcium and barium; most commonly, the feldspars are considered as solid solutions of three limiting compounds, \( \text{NaAlSi}_3\text{O}_8 \), \( \text{KAlSi}_3\text{O}_8 \), and \( \text{CaAl}_2\text{Si}_2\text{O}_8 \), which are respectively known as soda feldspar, potash feldspar and lime feldspar. Natural deposits of feldspar are generally solid solutions of either the soda and potash feldspars or the soda and lime feldspars.

2. Mode of Occurrence
As the main constituent of the igneous rocks making up the earth's surface, feldspars are abundantly distributed; the chief commercial sources are found in pegmatite dikes associated with other pegmatite minerals such as quartz and the various micas as well as minor amounts of tourmaline, beryl, garnet, spodumene, pyrite and magnesite. Although pegmatite deposits are widely distributed geographically, feldspars sufficiently free from impurities and occurring in large mineable quantities are not commonly found. For many years it has been the practice to separate the feldspar minerals from the associated impurities by a method of hand selection, but recent technological developments have led to flotation methods for the separation of quartz and mica. The flotation process has proved to be an effective method of producing large quantities of feldspar relatively free of undesirable impurities.

3. Geographic Occurrence
Commercial sources of feldspar are found in all the states of the Appalachian Region from Georgia to New York and in the New England States. South Dakota, Colorado, Minnesota, Arizona, California, Nevada, New Mexico and Texas also contain deposits. In recent years, North Carolina has been the chief feldspar-producing state, followed by Colorado, Virginia and South Dakota. In Canada, pegmatites containing commercial feldspar are found in the provinces from Nova Scotia and Labrador to Manitoba, and through the Rocky Mountain area.

In Europe, Sweden and Norway are the most important sources of feldspar; the Norwegian deposits are noted for their high purity. Great Britain does not have any appreciable amounts of pure feldspar although British potteries use a feldspathic material known as "Cornish Stone" which is a type of decomposed granite containing feldspar and quartz with varying amounts of kaolin, muscovite, fluorite, and topaz. Other feldspar deposits in Europe are found in Czechoslovakia, Germany, France, Italy, Rumania, Russia, and Finland. Elsewhere in the world, pegmatite deposits are located in China, India, Japan, Australia, New Zealand, Egypt, South Africa, and Argentina.

4. Commercial Use
The chief commercial value of the feldspars is in their use by the ceramic industries for the manufacture of glass, whiteware, and porcelain enamel products. Their fundamental characteristics and their behavior in the presence of other constituents are important considerations in the formulation of ceramic compositions. The following discussions will attempt to describe the basic features of feldspars and to show their behavior and influence when used as fluxing agents in whiteware bodies.

5. Acknowledgments
Funds for this project were furnished under a cooperative arrangement with the Engineering Experiment Station by the Consolidated Feldspar Corporation, which became the Consolidated Feldspar Department of International Minerals and Chemical Company. This work has been carried out under the general administrative direction of Dean W. L. Everitt, Director of the Engineering Experiment Station and Professor A. I. Andrews, Head of the Department of Ceramic Engineering.
II. FUNDAMENTAL PROPERTIES

6. Chemical Composition

In a consideration of the properties of feldspars, the chemical composition is of primary importance. The chemical analysis is an important key to the behavior of the material under various conditions and is the criterion most often used in evaluating the use of a particular feldspar in a whiteware body. The theoretical chemical compositions for the three major feldspars in their pure state are shown in Table 1.

Table 1

<table>
<thead>
<tr>
<th>Theoretical Compositions of Pure Feldspars*</th>
<th>K2O</th>
<th>Na2O</th>
<th>CaO</th>
<th>Al2O3</th>
<th>SiO2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Potash Feldspar</td>
<td>16.9</td>
<td>18.3</td>
<td>64.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>K2O·Al2O3·6SiO2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Soda Feldspar</td>
<td>11.8</td>
<td>19.4</td>
<td>68.8</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Na2O·Al2O3·6SiO2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Lime Feldspar</td>
<td>20.2</td>
<td>36.6</td>
<td>43.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CaO·Al2O3·2SiO2</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>* Weight percent.</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Since deposits of pure feldspar are rare or nonexistent, the naturally occurring minerals will have compositions which deviate somewhat from those shown in the table. The range of chemical compositions of typical commercial feldspars in the United States is shown in Table 2. The most significant variations in composition are found to be in the contents of SiO2, Al2O3, Na2O and K2O; therefore, it may be expected that these oxides will contribute to the variability of behavior of different feldspars. Although the amounts of CaO, MgO and Fe2O3 are small in relation to the other constituents, their effects on the thermal behavior are quite pronounced.

The chemical compositions of feldspars and other body materials have been used to estimate the quantities of ceramic bond in fired bodies and to differentiate the various phase constituents formed in the firing process. This method proposes to refer the chemical composition of the body to appropriate equilibrium diagrams and to relate the calculated phases to the fired physical properties of the body.

The conventional wet methods of chemical analysis are still widely employed for the determination of feldspar composition; however, recent instrumental procedures have been introduced for the analyses of silicates and have proven to be more rapid than the older methods and to give results of highly reproducible accuracy. Two of the instrumental methods particularly applicable for the analysis of feldspars are colorimetry and flame photometry. Colorimetric methods may be employed for the complete feldspar analysis with excellent reproducibility. Flame photometry has been used mostly for the determination of the alkali and alkaline earth metals, although complete silicate analysis methods are proposed.

Several proposals have been made for the classification of commercial feldspars on the basis of their chemical composition, physical properties and fineness of grind. An early basis for their classification and standardization was the alkali content and fusion temperature. Another system for commercial standards of quality was introduced by the Bureau of Standards to cover the specifications of ground feldspar used in ceramic processes. This method of classification was based on the particle size, chemical composition and end use of the material.

Although the proposed methods of feldspar classification were steps forward in the standardization of materials, their utilization was not fully realized. This lack of realization was no doubt largely due to the advent of feldspar blending. The introduction of blending and chemical control resulted in promoting standardization of composition and enabled the producers to supply large quantities of uniform material.

* Parenthesized superscripts refer to correspondingly numbered entries in the Bibliography.
While feldspar blending is chiefly based on the uniform control of chemical composition, the mode of occurrence and the inherent nature of each feldspar are also important considerations in judging the feasibility of blending.(18)

7. Mineralogical Composition

The mineralogical compositions of feldspars may often be determined by calculation from the chemical analysis to an accuracy suitable for certain applications such as those concerned with the free silica content. Since commercial feldspars consist essentially of microcline, albite and anorthite mixtures with some associated quartz, muscovite and kaolinite, the mineral composition may be calculated on a 6-component basis. This method has been simplified to such an extent that mathematical formulæ are available for direct computation of the mineral content. Formulae for the calculation are shown below.(19)

Percent Albite = 8.458E
Percent Anorthite = 4.960D
Percent Muscovite = 7.813C - 22.097A - 14.201D - 7.751E - 5.101F
Percent Quartz = B + 1.178C - 6.666A - 4.284D - 7.511E - 5.101F

where:
A = percent H₂O; B = percent SiO₂; C = percent Al₂O₃; D = percent CaO; E = percent Na₂O; F = percent K₂O, obtained from chemical analysis.

The mineralogical compositions of feldspars may be determined by direct methods such as optical and X-ray analyses, but the complex nature of feldspathic crystallization makes a review of the optical and X-ray methods beyond the scope of this discussion. Nevertheless, recent studies have greatly simplified these methods and are noteworthy of mention. In the soda-potash feldspar series a linear relationship has been found between the spacing of the (201) planes and the weight composition of the end members in solid solution. (20) This relationship is illustrated by Fig. 1. The relations between the refractive indices and the anorthite content of plagioclases have also been simplified and a mathematical expression has been proposed which describes the relation in the ranges of 0-30 percent and 64-100 percent anorthite. This is shown in Fig. 2. (21)

8. Structure

As the most important group of rock minerals, the geology and mineralogy of feldspars have long been the subjects of extensive study. Although there is probably more information regarding feldspars than any other group of minerals, they are not completely understood. Optical methods have been developed for their characterization, but the relationships between optical properties and chemical composition are yet to be definitely determined. Some of the optical properties are understood on the basis of crystal structure, and certain fundamental features have been established, even though a detailed knowledge of the complete structure is yet unknown.

All feldspars consist of a three-dimensional network of [SiO₄] and [AlO₄] tetrahedra in which all the tetrahedra share their oxygen atoms with their neighbors.(12) The fundamental units of [SiO₄] and [AlO₄] are linked together in a four-ring framework consisting of tetragons and collapsed octagons as viewed along the crystallographic a-axis. Positively charged ions of sodium, potassium or calcium are situated in the octagonal interstices of the negatively charged framework.(13) The fundamental structure of these tetrahedra is elastic to some degree and can adjust itself to the sizes of the cations in the octagonal openings. The crystal symmetry of the feldspars is dependent upon the size of these cations; relatively large cations such
as K⁺ give a symmetry which is monoclinic or nearly monoclinic while the small cations, such as Na⁺ and Ca⁺, cause a slight distortion of the structure and triclinic symmetry results.¹⁴

Commercial feldspars are considered on the basis of a three-component system, the end members of which are KAlSi₃O₈, NaAlSi₃O₈ and CaAl₂Si₂O₈, or potash, soda and lime feldspars, respectively. The extent of solid solution between the components and the manner in which the solid solutions are affected by the conditions which prevail during and after their formation remains partially unsolved. The potash and soda feldspars and their solid solutions are grouped together under the designation of alkali feldspars to set them off from the soda-lime feldspar solid solutions which are known as the plagioclases. No solid solution series exists between the potash and lime members since their mutual miscibility is practically zero at all temperatures.³

The feldspars may be mineralogically classified on the basis of their crystal symmetry as follows:

A. Monoclinic or nearly (pseudo-) monoclinic—
   Orthoclase KAlSi₃O₈
   Microcline KAlSi₃O₈
   Soda Orthoclase (KNa) AlSi₃O₈

B. Triclinic—
   Anorthoclase (NaK) AlSi₃O₈
   Albite NaAlSi₃O₈
   Anorthite CaAl₂Si₂O₈

The structural relationships between orthoclase and microcline are not completely known. The existence of dead bonds between some O²⁻ and K⁺ ions in orthoclase has been determined so that now it is generally believed that the difference between microcline and orthoclase is one of difference in atomic arrangement;¹⁵ it is assumed that in orthoclase the aluminum and silicon are randomly distributed, while in microcline these elements are considered to be in a particular set of lattice positions.

A potash feldspar approaching the composition of KAlSi₃O₈ is rarely found in commercial deposits; the orthoclase and microcline are always found to contain some soda feldspar. When relatively large amounts of soda feldspar are present, these feldspars are referred to as soda orthoclase or soda microcline. The molecules of KAlSi₃O₈ and NaAlSi₃O₈ form a continuous series of solid solutions at high temperatures, but at temperatures below 600 deg C there is a gap in the isomorphic series. At these lower temperatures, the solid solutions between potash and soda feldspars are metastable and under conditions of slow cooling show alterations into an oriented growth of sub-parallel lamellae which are alternately rich in soda and potash feldspar. Such intergrowths are called perthites or antiperthites. In the perthites, potash feldspar is the more abundant mineral, with soda feldspar occurring as uniformly-oriented films, veins or patches. In the antiperthites the soda feldspar is the more abundant mineral with potash feldspar interspersed.

Upon heating the perthites at 1000 deg C for several hundred hours, a homogeneous material will result. The mineral nature of feldspar is therefore dependent upon the temperatures of the magma from which the feldspar crystallized. Data have been obtained on the crystallization of one and two feldspar fields in the soda-potash feldspar system.¹⁶ Figure 3 illustrates the conditions under which soda-potash feldspars will crystallize as a single or a mixed feldspar. Any point on the curve dividing the two-feldspar field from the one-feldspar field represents the minimum temperature at which a feldspar of that composition will remain in stable equilibrium; if equilibrium is maintained below that temperature, unmixing will occur.

The soda and lime feldspar series has long been regarded as the example of an ideal isomorphic
series. This system, known as the plagioclase series, has been customarily designated as mixtures of different albite and anorthite ratios as follows:

<table>
<thead>
<tr>
<th>Mineral Name</th>
<th>Molecular Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Percent Albite</td>
</tr>
<tr>
<td>Albite</td>
<td>90-100</td>
</tr>
<tr>
<td>Oligoclase</td>
<td>70-90</td>
</tr>
<tr>
<td>Andesine</td>
<td>50-70</td>
</tr>
<tr>
<td>Labradorite</td>
<td>30-50</td>
</tr>
<tr>
<td>Bytownite</td>
<td>10-30</td>
</tr>
<tr>
<td>Anorthite</td>
<td>0-10</td>
</tr>
</tbody>
</table>

Plagioclases crystallized at high temperatures exhibit the perfect solid solution characteristics attributed to this system; however, there is evidence that in natural plagioclases there is a considerable miscibility gap between 30 and 70 mole percent anorthite. This gap is apparently affected by an albite inversion at about 700 deg C as shown in Fig. 4. Thus, although all appearances would indicate a perfect isomorphism in the plagioclases, and unmixing may take place during the cooling of the magmatic crystals and result in a mixture of highly ordered end members at room temperatures.

9. Thermal Properties

The melting characteristics of the soda, potash and lime feldspars have been determined for the pure materials and for their mixtures. It should be noted that the melting temperatures of feldspars are extremely difficult to obtain since the melting phenomena are very sluggish. The melting begins at the surfaces of crystals and proceeds so slowly that much of the crystal can exist in the presence of the melt for long periods of time even though the temperature is somewhat above that of the melting point. The melting point is considered as the temperature at which the crystal and the melt may exist in equilibrium and is determined by locating the temperature above which crystals show
melting tendencies and below which the crystals tend to grow. The exact temperature of this equilibrium is not easily determined and it has been necessary to express feldspar melting points in terms of temperature ranges.

Soda feldspar has been found to melt congruently at a very viscous liquid at a temperature of $1118 \pm 3$ deg C.\(^{(15, 18)}\) From the phase equilibrium diagram of the system $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$, as shown in Fig. 5a, a binary mixture of pure soda feldspar and silica is found to have a minimum melting temperature of $1062 \pm 3$ deg C (I); this mixture is equivalent to 68.5 percent soda feldspar and 31.5 percent silica. A ternary mixture of 66.0 percent soda feldspar, 33.3 percent silica and 0.7 percent alumina is shown to form an eutectic at $1050 \pm 10$ deg C (M).

Potash feldspar has been established as melting incongruently at $1150 \pm 20$ deg C to form crystals of leucite ($K_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$) and a viscous liquid (12.5 percent $K_2\text{O}$, 13.5 percent $\text{Al}_2\text{O}_3$, 74.0 percent $\text{SiO}_2$) which is more siliceous than the feldspar.\(^{(19)}\) With potash feldspar there is a long temperature interval during which leucite and the liquid may coexist at equilibrium; above temperatures of 1530 deg C the leucite crystals disappear. As a compound in the system $K_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$ (Fig. 5b), potash feldspar theoretically forms a binary eutectic with silica at $990 \pm 20$ deg C (I) when the composition is 58.0 percent potash feldspar and 42.0 percent silica; a ternary mixture of 56.2 percent potash feldspar, 43.2 percent silica and 0.6 percent alumina forms a theoretical eutectic at $985 \pm 20$ deg C (M).

The melting relations of binary mixtures of the feldspars have also been determined. In the soda feldspar-potash feldspar binary system as shown in Fig. 3 a minimum melting temperature of $1063 \pm 30$ deg C is obtained at three compositions (60, 65 and 70 percent of soda feldspar), but the composition of 65 percent soda feldspar and 35 percent potash feldspar is generally considered as the low melting mixture.\(^{(22)}\)

In the plagioclase series (Fig. 4), the soda and lime feldspars form a series of solid solutions with a continuous rise in the liquidus and solidus temperatures from pure soda feldspar to pure lime feldspar.

A recent study has been made to determine the nature of the liquidus surface of the ternary system soda feldspar-potash feldspar-lime feldspar.\(^{(23)}\) The data obtained to date has been concerned with the completion of melting; however, determinations are being made for the beginning of liquid formation.

The melting behaviors which have been discussed above are those of the pure feldspars and their mixtures under equilibrium conditions; the thermal relations of commercial feldspars, such as those used for the manufacture of whitewares, may be expected to be somewhat different due to the influence of impurities. In ceramic processes, equilibrium states are seldom achieved although the reactions taking place tend to approach those states; therefore, the rates at which the reactions proceed toward equilibrium must be considered.

It has been stated that feldspar melts approach equilibrium conditions very sluggishly due to their high viscosities. These viscosities of melts have been the basis of evaluating the thermal behavior of feldspars for ceramic use; it is a common practice to express this behavior in terms of fusibility or the plastic deformation of a feldspar cone when heated at a specified rate (i.e. 20 deg C per hr). When the feldspar cone deformation is compared to the deformation of standard pyrometric cones heated at the same rate, the fusibility is generally expressed in terms of pyrometric cone equivalents (p.c.e.).

At equivalent temperatures, the viscosities of soda feldspar melts have been found to be lower than the viscosities of potash feldspar melts; the mixtures of these alkali feldspars having intermediate values.\(^{(24)}\) Accordingly, the p.c.e. ranges of typical feldspars used in whiteware compositions are:\(^{(25)}\)

- **Cone 4-5(1165-1180 deg C)** for high soda feldspars
- **Cone 5-8(1180-1225 deg C)** for intermediate alkali feldspars
- **Cone 8-10(1225-1260 deg C)** for high potash feldspars

Binary mixtures of 65 parts soda feldspar and 35 parts potash feldspar have been found to deform at temperatures slightly below the deformation temperature of the soda feldspar.\(^{(26)}\) Ternary mixtures consisting of 70 parts soda feldspar, 25 parts potash feldspar and 5 parts lime feldspar give a lower deformation temperature than any other feldspar mixture.\(^{(27)}\)

Viscosity studies have shown that the presence of uncombined $\text{SiO}_2$ will increase the viscosity of...
Fig. 5. Portions of the Equilibrium Diagrams, Soda-Alumina-Silica and Potash-Alumina-Silica
a feldspar melt and also deter the drop in viscosity with increasing temperatures.\textsuperscript{(25)} Accordingly, the presence of free SiO$_2$ increases the p.c.e. value of feldspars. This effect is most pronounced in high soda feldspars; the presence of 10 percent of uncombined SiO$_2$ in a soda feldspar is sufficient to increase its refractoriness, whereas a high potash feldspar may tolerate up to 20 percent of free SiO$_2$ before the p.c.e. is materially increased.\textsuperscript{(25)}

Amounts of Fe$_2$O$_3$ in the order of 0.3 percent are sufficient to lower the viscosity of a feldspar melt and thereby lower its p.c.e. value. The addition of the oxides of Ca, Mg, Ba and Zr in amounts of 2 to 5 percent have also been found to lower the viscosities of feldspars melts and reduce the deformation temperatures.\textsuperscript{(28, 30)}

Studies of binary systems of feldspars and clay have not revealed any deformation eutectics; however, it has been established that as a feldspar melts it takes the decomposition products of clay into solution at a rate dependent upon the temperature and the surface areas. In general, it has been shown that clay is more soluble in soda feldspar than in potash feldspar.\textsuperscript{(31, 32)}

The relative solubilities of clay and quartz in feldspar melts have not been definitely established; however, it is known the presence of both materials affects their mutual solubility; as the quartz content is increased the solubility of clay in the feldspar melt is diminished.\textsuperscript{(33)}

The thermal expansion coefficients of feldspar glasses may be predicted from the expansion factors proposed by Hall\textsuperscript{(34)} if the free quartz content is not in excess of 4 percent.\textsuperscript{(35)} In the crystalline state soda feldspars have a higher coefficient of expansion than potash feldspars; in the fused state the thermal expansion of the fused potash feldspar is considerably greater than that of fused soda feldspar chiefly due to the formation of leucite in the potash feldspar melt. The thermal expansion of fused potash feldspar is effectively reduced by the presence of free SiO$_2$ or soda feldspar due to a greater solution of the leucite.

The density of feldspar is reduced when it changes from the crystalline to the fused state. A high soda feldspar with a density of 2.635 in its raw state will fuse to a density of 2.37, and as a result will occupy a 12 percent greater volume. Similarly a high potash feldspar of 2.572 density will fuse to a density of 2.37 with a volume increase of 9 percent.\textsuperscript{(25)}

The thermal properties of feldspars have been the subject of considerable study in the past and will doubtless be the object of many future investigations. Several factors have been established as significant for the thermal behavior of feldspars and may be summarized as:

(a) chemical composition, which determines the ultimate equilibrium condition for any specified temperature

(b) mineralogical composition, which determines the initial point from which reactions will proceed and the nature of melting (i.e. congruent or incongruent)

(c) particle size of the mineral constituents, which determines the extent of surface area and the rate at which melting will take place

(d) viscosity of the melt formed, which ultimately determines the rate at which reactions will proceed toward the equilibrium state.\textsuperscript{(36)}

10. Solubility in Water

In addition to the thermal behavior of feldspars, the solubility of feldspars in water is of interest in ceramic processes. No definite conclusion has been made as to whether a high soda or a high potash feldspar is more soluble in water. It is well known that when finer ground fractions of feldspar are placed in water, decomposition takes place and alkalies are extracted from the feldspar. The alkalinity of the water is immediately increased and continues to increase with time although the rate of increase gradually becomes smaller with longer exposure.\textsuperscript{(37)}
III. USE IN WHITEWARE COMPOSITIONS

11. Physico-Chemical Behavior

The function of feldspar in whiteware bodies is that of a flux and as such it takes part in physico-chemical reactions with other crystalline phases. The old conception that the feldspar serves as a bond for the crystalline phases is being rejected and current theories consider that the bonding of grains and formation of a dense mass is due to a deep inter-diffusion of phases.\(^3\)

During the firing of a whiteware body composed of feldspar, clay and flint, the first glassy phase to form is due to a ternary eutectic. With pure materials and equilibrium conditions the temperature of the eutectic formation would be \(990 \pm 20\) deg C with a pure potash feldspar or \(1050 \pm 10\) deg C with a pure soda feldspar, and the amount of melt formed would vary with the amount of feldspar present. In bodies which contain somewhat impure materials, the eutectic temperatures may be somewhat lower. Commercial bodies are fired at a rate which is necessarily much too rapid for the achievement of equilibrium conditions; and while some eutectic formation may occur at the theoretical temperature, the amount of glass formed at that point will be very minute. The eutectic formation may be more readily detected at somewhat higher temperatures. Partial fusion has been observed at \(1075 - 1085\) deg C in bodies fired at a rate of \(10\) deg C per minute.\(^3\) As the temperature is increased more liquid melt is formed which begins to draw particles together by surface tension and progressive solution takes place. Mullite, which was formed from the decomposition of clay, may diffuse into the melt or the crystals may continue to grow at higher temperatures and increased time of heat treatment. Mullite is also formed by recrystallization as the melt becomes saturated or when the temperature is lowered. Above the normal vitrification range, air, which had been entrapped within the pores of the body by the melt, will build up sufficient pressure to expand against the viscous glass and cause bloating or blistering.

The fluxing influence of a feldspar used in a body does not necessarily follow the order anticipated by the fusion behavior of the feldspar alone or combinations of the feldspar with one of the other body ingredients. Each body composition is an individual system and its thermal reactions are dependent on all of the components collectively. It might be expected that a body containing a high soda feldspar of low p.c.e. value would reach maturity at a temperature considerably lower than a similar body containing a high potash feldspar of high p.c.e. value; actually, the difference in the maturing temperatures of the two bodies may be of small magnitude (i.e. 10 deg C) in contrast to the difference in fusion temperatures (i.e. 70 deg C) of the individual feldspars. It has been shown that bodies containing potash feldspar may even mature before those containing equivalent amounts of soda feldspar if free calcia is present in the compositions.\(^3\)

It has been generally accepted that finer particle sizes of feldspar and flint lower the maturing temperatures of feldspar-flint-clay bodies and that only the finest particles of feldspar form a glassy matrix. The larger feldspar particles fuse and become isotropic but do not lose their original shape to any great degree.\(^4\) The advantage of finer particle size is most effectively realized with controlled firing procedures; a more gradual heating rate or longer soaking time at the maturing temperature will effect a more representative saturation of the glassy phase.\(^4\) A body which achieves a porosity of zero percent in 10 min at 1250 deg C may reach the same degree of vitrification at 1200 deg C in a longer time period.\(^4\)

In addition to the effect of finer particle size on the maturing temperature of bodies, the fineness also governs the degree of firing shrinkage, strength, thermal expansion and warpage. Finer grinds of feldspar tend to increase the firing shrinkage and strength; however, greater warpage and lower thermal expansion result.\(^4\) The warpage of bodies is a function of the viscosity and the proportion of glassy phase formed at elevated temperatures. Greatest warpage is obtained in bodies of high flint and high feldspar contents; increasing clay
content at the expense of flint effectively reduces warpage.\textsuperscript{45, 46} Soda feldspars which form less viscous glasses have been found to cause more body warpage than potash feldspars. The thermal expansion of fired bodies is largely dependent upon the amount of uncombined quartz and will therefore decrease with higher firing temperatures.\textsuperscript{47}

In general, higher thermal expansions have been obtained in bodies containing soda feldspars.\textsuperscript{125}

12. Purpose and Scope of Experimental Investigation

A laboratory investigation was conducted to determine the comparative effects of high potash and intermediate soda-potash feldspars on the fired properties of various types of whiteware bodies. The need for this study has been accentuated by the rapid depletion of readily available supplies of high grade potash feldspar. In the past, feldspars of highest potash content have been sought as the best type for use in ceramic whitewares, but recent studies have shown that feldspars of considerable soda content may be used advantageously as replacements for very high potash feldspars with little or no significant changes in the fired properties of whiteware bodies.\textsuperscript{48}

Four commercial feldspars were used in the studies of bodies. The body compositions selected for the investigation were those which are typical of:

(a) semi-vitreous dinnerware
(b) hotel china
(c) sanitary ware
(d) electrical porcelain
(e) floor tile

Compositions were formulated to study the effects of different types of feldspars, variable feldspar content and the effect of auxiliary fluxes in combination with the feldspars.

Bodies were prepared and formed into test specimens according to commercial processing methods. The details of body preparation, specimen formation, firing and testing procedures are described in the Appendix.

13. Properties of Feldspars Used

The properties of the four commercial feldspars were determined and are shown in Table 3. The feldspars Buckingham and Custer are of the high potash type with $\text{K}_2\text{O}$ to $\text{Na}_2\text{O}$ ratios of more than 3 to 1 and microcline to albite ratios greater than 2 to 1. These feldspars may be expected to give somewhat similar firing properties in typical feldspar-flint-clay bodies due to their high potash content. A-3 and F-4 feldspars have $\text{K}_2\text{O}$ to $\text{Na}_2\text{O}$ ratios of 1.53 and 1.07 respectively, so that they are classified as intermediate soda-potash feldspars.

Actually, F-4 may be considered as a plagioclase-potash feldspar due to its appreciable anorthite content. It also should be noted that A-3 contains a considerable amount of free quartz and therefore the actual feldspathic content per unit weight is somewhat reduced. F-4 is a flotation-processed feldspar which is relatively free of the associated minerals such as muscovite.

The chemical analyses of the feldspars are those furnished by the Consolidated Feldspar Department of the International Minerals and Chemical Corporation. The mineralogical compositions were calculated from these analyses according to the methods outlined by Koenig.\textsuperscript{9}

The particle size distribution was determined by the Andreasen Pipette method as proposed by Loomis\textsuperscript{490} and Russell and Weisz,\textsuperscript{50} and is presented in tabular form.

Fusibilities of the individual feldspars were determined by firing several small slender trihedral

| Table 3
<table>
<thead>
<tr>
<th>Properties of Feldspars Studied</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. Chemical Analyses</td>
</tr>
<tr>
<td>Weight Percent</td>
</tr>
<tr>
<td>Concept</td>
</tr>
<tr>
<td>Na$_2$O</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
</tr>
<tr>
<td>CaO</td>
</tr>
<tr>
<td>MgO</td>
</tr>
<tr>
<td>Na$_2$O</td>
</tr>
<tr>
<td>K$_2$O</td>
</tr>
<tr>
<td>Ignition Loss</td>
</tr>
<tr>
<td>b. Mineralogical Analyses (calculated weight percent)</td>
</tr>
<tr>
<td>Minerals</td>
</tr>
<tr>
<td>Albite</td>
</tr>
<tr>
<td>Microcline</td>
</tr>
<tr>
<td>Anorthite</td>
</tr>
<tr>
<td>Free Quartz</td>
</tr>
<tr>
<td>Muscovite</td>
</tr>
<tr>
<td>Kaolinite and Adj. Minerals</td>
</tr>
<tr>
<td>c. Particle Size Distribution by Sedimentation Analyses (weight percent)</td>
</tr>
<tr>
<td>Less than 30 microns</td>
</tr>
<tr>
<td>Less than 20 microns</td>
</tr>
<tr>
<td>Less than 15 microns</td>
</tr>
<tr>
<td>Less than 10 microns</td>
</tr>
<tr>
<td>Less than 5 microns</td>
</tr>
<tr>
<td>Less than 2 microns</td>
</tr>
<tr>
<td>d. Fusibility (p.e.c. heating rate 10° deg F per hr)</td>
</tr>
<tr>
<td>Na$_2$O</td>
</tr>
<tr>
<td>K$_2$O</td>
</tr>
<tr>
<td>Total K$_2$O</td>
</tr>
</tbody>
</table>

* After 24 hrs of exposure: 1 part by weight of feldspar to 25 parts by weight of distilled water; determined by flame photometer.
pyramids of each at a heating rate of approximately 50 deg F per hr in an electric furnace and observing their deformation in relation to the deformation of standard pyrometric cones which were fired simultaneously. The reported values of fusibility are expressed in terms of p.c.e. and represent the pyrometric cone equivalent at the temperature when each feldspar specimen had deformed such that the tip of the specimen was level with the supporting plaque.

The relative solubilities of the feldspars in water were obtained using particle sizes between 44 and 53 microns. Ten grams of each feldspar, collected between Nos. 270 and 325 mesh sieves, were placed in 300 ml pyrex bottles containing 250 ml of double distilled water. The bottles were sealed and tumbled end over end for 24 hr; after the agitation period the feldspar water mixtures were centrifuged and the supernatant liquid drawn off for solubility tests. Ninety ml of each liquid were added to 10 ml of a standard Li solution. These solutions were examined with a Perkin-Elmer Flame Photometer for NaO and KO content. The values expressed in the table of solubility represent the milliequivalents of NaO and KO per liter taken into solution from each feldspar after 24 hr of exposure in water.

14. Semi-Vitreous Ware

Composition

Four series of compositions were formulated for the purpose of studying the fired characteristics of typical semi-vitreous dinnerware bodies as influenced by high potash and intermediate soda-potash type feldspars, varying feldspar content, and combination of feldspar with small amounts of auxiliary fluxes. A total of 21 bodies was studied; the compositions are shown in Table 4.

A typical semi-vitreous body which was known to achieve a porosity of approximately 10 percent at cone 10 was selected for a base composition. This composition contained:

- 33.5 percent of flint
- 36.0 percent of mixed Tennessee and Kentucky ball clays
- 21.0 percent of mixed Georgia and North Carolina kaolins
- 13.5 percent feldspar

Bodies of Series I (Table 4) were of this composition; succeeding series of bodies represent variations in the base composition such as increased feldspar content, addition of auxiliary flux, or both. The deviations from the base composition were, for the most part, compensated for by appropriate alterations in the kaolin content.

Four commercial feldspars were used for the compositions of Series I; the body in which each was contained may be readily identified by the code letter following the body composition number. Thus, bodies SV1A and SV1C respectively contained Buckingham and Custer feldspars, which are of the high potash type; bodies SV1A and SV1F contained A-3 and F-4 feldspars respectively. These feldspars may be considered as intermediate soda-potash types. The analyses and properties of the feldspars used have been shown in the previous section. The Custer (C) and F-4 (F) feldspars were selected as representative of the high potash and intermediate soda-potash type feldspars and were used for comparative purposes.

Bodies of Series II represent increases in the feldspar content over that of the base composition. Increases to 15.0 and 16.5 percent feldspar were made at the expense of Pioneer kaolin. Series III was composed of bodies in which 2.0 percent additions of various fluxes were added to the base composition.

Table 4

<table>
<thead>
<tr>
<th>Body No.</th>
<th>Series I</th>
<th>Series II</th>
<th>Series III</th>
<th>Series IV</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1A</td>
<td>1B</td>
<td>1C</td>
<td>1F</td>
</tr>
<tr>
<td>Feldspar A</td>
<td>13.5</td>
<td>13.5</td>
<td>15.0</td>
<td>16.5</td>
</tr>
<tr>
<td>Feldspar B</td>
<td></td>
<td>13.5</td>
<td>15.0</td>
<td>16.5</td>
</tr>
<tr>
<td>Feldspar C</td>
<td></td>
<td></td>
<td>13.5</td>
<td>13.5</td>
</tr>
<tr>
<td>Feldspar F</td>
<td></td>
<td></td>
<td>13.5</td>
<td>13.5</td>
</tr>
<tr>
<td>C &amp; C Ball Clay</td>
<td>7.0</td>
<td>7.0</td>
<td>7.0</td>
<td>7.0</td>
</tr>
<tr>
<td>Imperial Ball Clay</td>
<td>5.0</td>
<td>5.0</td>
<td>5.0</td>
<td>5.0</td>
</tr>
<tr>
<td>Terra No. 2 Ball Clay</td>
<td>7.0</td>
<td>7.0</td>
<td>7.0</td>
<td>7.0</td>
</tr>
<tr>
<td>Old Mine 4 Ball Clay</td>
<td>7.0</td>
<td>7.0</td>
<td>7.0</td>
<td>7.0</td>
</tr>
<tr>
<td>Pioneer Kaolin</td>
<td>12.0</td>
<td>12.0</td>
<td>12.0</td>
<td>12.0</td>
</tr>
<tr>
<td>Kemco Kaolin</td>
<td>9.0</td>
<td>9.0</td>
<td>9.0</td>
<td>9.0</td>
</tr>
<tr>
<td>E.P.K.</td>
<td>6.0</td>
<td>6.0</td>
<td>6.0</td>
<td>6.0</td>
</tr>
<tr>
<td>Ottawa Flint</td>
<td>33.5</td>
<td>33.5</td>
<td>33.5</td>
<td>33.5</td>
</tr>
<tr>
<td>Whiting</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
<td>2.0</td>
</tr>
</tbody>
</table>
compositions. The fluxes used were whiting, low lime talc, tremolitic talc and magnesite.

In Series IV only F-4 feldspar was used in the body compositions for the purpose of determining the effects of varying combinations of feldspar with tremolitic talc. These compositions contained 15.0 and 16.5 percent F-4 feldspar in combination with 2.0 percent tremolitic talc, and combinations of 13.5, 15.0 and 16.5 percent F-4 feldspar with 4.0 percent tremolitic talc.

Processing and Firing

The semi-vitreous dinnerware bodies were prepared by buffing and filter pressing. Test specimens consisted of extruded \( \frac{3}{4} \) in. diam rods. Sets of 15 specimens of each body composition were fired to temperatures of 2140 deg F, 2200 deg F, 2230 deg F, 2280 deg F and 2340 deg F in an electrically heated kiln. These temperatures corresponded to cone equivalents of 5\(^*\), 7\(^*\), 8\(^*\), 9\(^*\), and 11\(^*\). The temperature rise was controlled at approximately 100 deg F per hr up to 2000 deg F and 50 deg F per hr thereafter until the peak temperature was reached and held for 11/2 hr. In addition to the laboratory firings, several bodies were fired in a commercial tunnel kiln to cone 9\(^*\) (See Table 5).

Tests were conducted on the fired specimens to obtain the fired porosity, modulus of rupture and volume shrinkage. The thermal expansion and crystalline nature of bodies fired to cone 9\(^*\) were also determined.†

Results

Physical Properties. The physical properties of the fired semi-vitreous dinnerware bodies are presented in Fig. 6. The data in this form are convenient for examining the relations between the firing temperatures and the changes in the properties of each body; however, a comparison of the properties of all bodies at some common condition such as maturity is more desirable. For convenience, therefore, the authors have arbitrarily assumed maturity in the semi-vitreous bodies to be that condition at which the body has a fired porosity of 10 percent. In order to obtain the body properties at the point of 10 percent porosity, the following procedure was followed: A vertical line is drawn through the 10 percent point on the porosity curve; the intersections of the vertical line with the temperature scale and the other physical property curves determine the maturing temperature and physical properties at that temperature. The results thus obtained with all bodies are shown as bar graphs in Fig. 7.

In compositions of Series I, it was observed that the bodies containing high potash feldspars (SV1B and SV1C) matured at about the same temperature with similar strengths and shrinkages. The soda-potash feldspar bodies (SV1A and SV1F) matured at slightly higher temperatures with less strength than the potash feldspar bodies. The shrinkage of SV1F was similar to the shrinkage of SV1B and SV1C, while SV1A showed less shrinkage, possibly due to the low free quartz content of A-3 feldspar.

Increasing feldspar content lowered the maturing temperature of the bodies somewhat as would be expected; however, it was observed that while body SV1F matured at a slightly higher temperature than SV1C, the two bodies, SV2F and SV2C, matured simultaneously, and SV3F matured earlier than SV3C. The increased feldspar contents caused no appreciable change in the strengths of the matured bodies. The shrinkages of bodies made with high potash feldspars were essentially unchanged when the feldspar content was increased from 13.5 to 16.5 percent; however, in the soda-potash bodies an increase in shrinkage was noted when the feldspar content was raised from 13.5 to 15.0 percent.

From the results of physical properties obtained with the first two series of compositions, it may be concluded that direct weight substitutions of soda-potash feldspar may be made for high potash feldspar in a semi-vitreous type body with (a) little or no change in maturing temperature, (b) slightly less transverse strength, and (c) little or no increase in shrinkage.

*The superscript refers to the degree of bending of the cone tip corresponding to the numerals on a clock face between 1 and 6.
† For detailed description of the equipment and procedures employed in the processing and testing of bodies, see Appendix.
Fig. 6. Fired Properties of Semi-Vitreous Bodies
Fig. 7. Properties of Semi-Vitreous Bodies at Maturity (10 percent Porosity)
The addition of 2 percent whiting to the base composition was effective in reducing the maturing temperature of the potash feldspar body (SV4C) to about one cone below that of the base body. The effect of a like whiting addition to the soda-potash body (SV4F) was not quite as pronounced. The whiting addition to the potash feldspar body resulted in reduced strength at maturity. Little change of matured shrinkage was noted.

The effect of 2 percent low lime talc with the potash feldspar was very similar to that obtained with whiting; with soda-potash feldspar slightly more fluxing was obtained but with little change in the matured properties. The addition of 2 percent tremolitic talc to the base composition was only slightly more effective in its fluxing action than the whiting or low lime talc; however, somewhat greater shrinkage resulted.

Bodies SV7C and SV7F contained 2 percent additions of magnesite. The maturing temperature of SV7C was about 2 cones lower than the base body SV1C, while SV7F matured about 3 cones lower than the base body SV1F. These marked reductions in maturing temperature were accompanied by increases in volume shrinkages but no appreciable changes in strength.

Combined increases in feldspar content and talc additions reduced the maturing temperature very markedly. A 3 percent increase in feldspar content and an addition of 4 percent of talc reduced the maturing temperature by 5 cones. At the lower maturing temperatures, slightly less strength and reduced shrinkages were obtained.

A comparison of the results obtained from a commercial firing and a laboratory kiln firing are shown in Table 5. It is noted that in all bodies the fired porosities obtained with the laboratory kiln were lower than those obtained from the commercial firing. This difference may be attributed to the difference in firing rates, the commercial rate being almost twice as rapid as the laboratory cycle. The values of strength obtained from the two firings were in close agreement.

**Crystalline Content.** The crystalline nature of the specimens fired to cone 9° were determined by X-ray analysis.* The only crystal phases identified in the bodies were quartz, mullite, and in some cases, cristobalite. The crystalline content of Series I bodies made with potash feldspar was entirely quartz and mullite; the soda-potash feldspar bodies showed a slight amount of cristobalite present but not in excess of 5 percent. Increasing feldspar content was found to decrease the amount of free quartz and to increase the mullite formation.

The addition of whiting also decreased the quartz content and slightly increased the mullite formation. A small degree of cristobalite formation was noted in the soda-potash body. In those bodies containing 2 percent of tale, the amount of free quartz was considerably reduced and some cristobalite development was noted in the potash feldspar bodies; while in the soda-potash bodies considerable cristobalite development occurred. Magnesite was found to cause extensive cristobalite formation with the soda-potash feldspars and to a lesser degree with high potash feldspars. The quantitative determination of cristobalite for several fired bodies is shown in Table 6.

<table>
<thead>
<tr>
<th>Body No.</th>
<th>Percent Feldspar</th>
<th>Percent Tremolite Talc</th>
<th>Percent Cristobalite (± 2 percent) After Firing to Cone 9°</th>
</tr>
</thead>
<tbody>
<tr>
<td>1C</td>
<td>13.5 C</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>1P</td>
<td>13.5 F</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>6C</td>
<td>13.5 C</td>
<td>2.0</td>
<td>12</td>
</tr>
<tr>
<td>6P</td>
<td>13.5 F</td>
<td>2.0</td>
<td>11</td>
</tr>
<tr>
<td>8F</td>
<td>15.0 F</td>
<td>2.0</td>
<td>10</td>
</tr>
<tr>
<td>9F</td>
<td>16.5 F</td>
<td>4.0</td>
<td>16</td>
</tr>
<tr>
<td>10F</td>
<td>13.5 F</td>
<td>4.0</td>
<td>13</td>
</tr>
<tr>
<td>11F</td>
<td>15.0 F</td>
<td>4.0</td>
<td>12</td>
</tr>
</tbody>
</table>

The compositions which showed high cristobalite formation were fired in an X-ray furnace to determine the temperature at which the cristobalite formed. The formation was found to occur during the cooling cycle at temperatures between 1100 deg C and 1000 deg C. The cristobalite was formed by the process of separation with crystal deposition of SiO2 or devitrification.

**Thermal Expansion.** The linear thermal expansion curves for bodies of Series I and II fired to cone 9°, are shown in Fig. 8. The expansions observed with the soda-potash feldspar bodies were slightly higher than those noted in bodies containing potash feldspar. The high expansion of body SV1A is undoubtedly due to the high free quartz content of A-3 feldspar. An increase of feldspar causes more crystalline silica to dissolve, thus decreasing the expansion.

The combination of auxiliary fluxes and potash feldspar caused a reduction in the thermal expansion of bodies fired to cone 9°. In bodies containing soda-potash feldspar, the addition of auxiliary fluxes which contained considerable percentages of

---

* See Appendix for description of X-ray unit and techniques employed.
MgO were found to cause a hump in the thermal expansion curves between 150—250 deg C, indicating the presence of cristobalite. The expansion data obtained with bodies containing the soda-potash feldspar and tremolitic talc are shown in Fig. 9. The most pronounced cristobalite inversion was noted in body SV10F which contained 13.5 percent of F-4 feldspar and 4 percent tremolitic talc. This data agrees favorably with the X-ray analysis since body SV10F was found to have the greatest cristobalite development among those bodies containing tremolitic talc as an auxiliary flux.

Thermal Shock: The influence of cristobalite on the thermal shock properties of the bodies was studied using glazed 6-in. coupe plates. Five plates of each of the compositions were heated to 480 deg F in an electric oven and quenched in a dye solution at 70 ± 2 deg F; the number of cycles required to cause a body or glaze defect was recorded. Table 7 shows selected results of the test. The bodies containing extensive cristobalite development were found to have a reduced thermal shock resistance although their crazing resistance was improved. Such bodies are likely to dent in the kilns unless special precautions for cooling are observed.

15. Hotel China

Composition

Two series of hotel china compositions were formulated as shown in Table 8. A typical composition containing 21 percent feldspar was selected as a base composition and four bodies of this type were made to show the influence of the four commercial feldspars, A-3, Buckingham, Custer and F-4, when each is used as the total flux content. These bodies are represented by compositions from 2A to 2F in Series I. All other bodies contain either Custer or F-4 feldspars.

Processing and Firing

The hotel china bodies were prepared by a process of ball milling and filter pressing. After one week of aging, the bodies were formed into 3/4-in. diam rods by extrusion. Specimens were fired in an electrically-heated kiln at a rate of approximately 75-80 deg F per hr up to 2000 deg F and 50 deg F per hr thereafter to temperatures of 2180, 2220,
2250, 2280, 2310, 2350, 2400, and 2450 deg F. A 1½ hr soak at these temperatures was used to deform standard pyrometric cones to 7°, 8°, 9°, 10°, 11°, 12°, 13°, and 13° respectively.

Results

The physical properties obtained with the fired specimens of hotel china bodies are shown in Fig. 10. In the bodies which contain 18 percent of either Custer or F-4 feldspar, vitrification was observed to occur at a temperature of approximately cone 12°, where the bodies developed zero porosity and a transverse strength in excess of 10,000 psi. The volume shrinkages of the two bodies were found to be similar with a value of approximately 30 percent at vitrification. Maximum values of strength and shrinkage were observed at cone 13°; the body containing Custer feldspar had slightly greater ultimate strength than the corresponding F-4 feldspar body.

With 21 percent feldspar (2A-2F), the firing behavior of bodies containing A-3, Buckingham, Custer and F-4 feldspars may be compared. The body containing A-3 feldspar was vitrified at cone 12°, while the bodies containing Buckingham, Custer or F-4 feldspar vitrified at a half cone lower, cone 12°. The potash feldspars promoted slightly more strength and somewhat less shrinkage than the soda-potash feldspars. The A-3 feldspar composition did not show evidence of overfiring at cone 13°, while the potash feldspars bodies indicated overfiring at this temperature. The body containing F-4 feldspar was found to begin overfiring at cone 13°.

Bodies which contained 25 percent of either Custer or F-4 feldspar were found to vitrify between cones 11° to 12° with evidences of overfiring at cone 13°. The F-4 composition approached vitrification at a more rapid rate than the Custer feldspar body and also showed a little more shrinkage.

The additions of talc to the hotel china bodies containing 21 percent feldspar increased the rate of vitrification, thus lowering the maturing temperature. An addition of 2 percent talc to the Custer feldspar body promoted vitrification at cone 11° which was one cone lower than the maturing temperature with no talc present. In a similar body containing F-4 feldspar, a 2 percent talc addition reduced the vitrification temperature from cone 12° to about cone 10°. The bodies containing 2 percent talc began to overfire at cone 12°. An increase in the talc addition to 4 percent was effective in promoting the vitrification of the Custer body at cone 10° and the F-4 body at about cone 9°. Overfiring took place at cone 12°. The bodies of increased feldspar content with 2 percent talc additions vitrified at cone 10° and overfired at cone 12°. In general it was observed that when talc was added, the rate of fluxing was more rapid in F-4 feldspar bodies than in corresponding Custer feldspar bodies, and as a result had shorter firing ranges.

The fluxing effect of whiting with Custer and F-4 feldspars was observed in bodies 6C and 6F. Two percent additions to bodies containing 21 percent of feldspar effected vitrification after firing to cone 12°; overfiring was noted at cone 13°. The whiting was found to be a more efficient flux with Custer feldspar than with F-4 feldspar.

A qualitative analysis of the crystalline phases present in the matured hotel china bodies investigated showed evidence of some cristobalite development in all bodies which contained F-4 feldspar, while little or no cristobalite was noted in Custer feldspar bodies.

16. Electrical Porcelain

Composition

One series of electrical porcelain-type bodies was investigated. The composition of this series of bodies is shown in Table 9; the only variation in the compositions of the four bodies is the type of feldspar used. As in previous studies, the four commercial feldspars, A-3, Buckingham, Custer

Table 8

<table>
<thead>
<tr>
<th>Composition of Hotel China Bodies</th>
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<tbody>
<tr>
<td></td>
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<tr>
<td></td>
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<tr>
<td></td>
</tr>
<tr>
<td>A-3 Feldspar</td>
</tr>
<tr>
<td>Buckingham Feldspar</td>
</tr>
<tr>
<td>Custer Feldspar</td>
</tr>
<tr>
<td>F-4 Feldspar</td>
</tr>
<tr>
<td>Ottawa Flint</td>
</tr>
<tr>
<td>Old Mine No. 4 Ball Clay</td>
</tr>
<tr>
<td>Kansas Kaolin</td>
</tr>
<tr>
<td>Pioneer Kaolin</td>
</tr>
<tr>
<td>Tenderlite No. 1 Whiting</td>
</tr>
</tbody>
</table>

21
35 35 35
8.5 8.5 8.5
14 14 14
12.5 12.5 12.5
9 9 9
6F 7C 7F
21
35
8.5
14
11.5
8
2 2
and F-4 are employed as the total feldspathic contents, and the bodies in which they are contained may be identified by the code letters following the composition designations.

Processing and Firing

The electrical porcelain bodies were prepared by blunging and filter pressing. Test specimens for shrinkage, porosity and modulus of rupture determinations were formed by extrusion. The test specimens consisted of 7/8-in. diam rods. Samples for the determination of dielectric properties were formed by plastic pressing in a plaster mold; these samples were made in the shape of a 6 3/4-in. diam disc 3/16 in. thick, the surfaces of which were ground approximately parallel after drying.
Test specimens were fired in an electrically heated kiln at a heating rate of approximately 80 deg F per hr to temperatures of 2230, 2260, 2300, 2340, 2375, and 2410 deg F and held at these temperatures for 2 hr. Cone plaques set among the specimens in each burn indicated the above firings to be respectively equivalent to cones 8°, 9°, 11°, 12°, 12°, and 13°. The dielectric test samples were fired to the vitrification temperatures determined for each body composition.

**Results**

The physical properties determined for the electrical porcelain bodies are shown in Fig. 11. Body EPIA, which contained A-3 feldspar was found to mature later than any of the other bodies showing highest values of porosity and lowest volume shrinkage throughout the firing range. As shown in other type bodies, this behavior was probably due to the high free quartz content of A-3 feldspar. The vitrification of body EPIA was shown to occur at a temperature between cones 12° and 13°.

The two potash feldspar bodies, EPIB and EPIC, had similar fired physical characteristics throughout the firing range studied. The vitrification of both of these bodies was complete at cone 12°-12°.

The body showing the earliest vitrification in this series of electrical porcelain bodies was EPIF which contained the flotation feldspar, F-4. This body was shown to achieve complete vitrification at temperatures between cone 11° to 12°.

The modulus of rupture values obtained with this series of bodies indicated the F-4 feldspar bodies gave higher strengths than the potash feldspar bodies at equivalent firing temperatures. The firing shrinkage of the F-4 feldspar body was greater than any of the other bodies in this series throughout the firing range.

The results of the electrical tests conducted on the dielectric samples are shown in Table 10. The measurements were made by the Locke Department of the General Electric Company. The electrical properties which were of chief concern in evaluating these bodies were the dielectric strength and loss factors. The dielectric strengths of conventional high voltage porcelains range between 250 and 300 volts per mil; all of the bodies tested show favor-
able dielectric strengths: the highest breakdown strength was shown by the Custer feldspar body and the lowest breakdown strength in the F-4 feldspar samples. The F-4 feldspar body also had the highest loss factor of the series.

17. Sanitary Ware Composition

In the study of sanitary ware bodies, four series of compositions were formulated as shown in Table 11. A typical sanitary ware composition containing 32 percent of feldspar was selected for the first series of compositions. This series consisted of four bodies, each of which was made with a different commercial feldspar — A-3, Buckingham, Custer and F-4. In Series II, 2 percent of tremolitic talc was added to each of the above bodies. The talc addition was increased to 4 percent in series III. In Series IV, bodies containing Custer and F-4 feldspars were made at a reduced feldspar level (28 percent) with and without a 2 percent talc addition. All variations from the base composition were compensated for by appropriate alterations in the kaolin content.

Processing, Casting and Firing

The sanitary ware bodies were prepared by blunging and filter pressing. After one week of aging, the casting characteristics of the various body compositions were determined at six electrolyte levels. These casting trials were determined with slip batches containing 1000 grams of dry body. The sodium carbonate content was held constant at 0.05 percent in all trial slips but the sodium silicate content was varied from 0.05 to 0.175 percent in increments of 0.025 percent.

The results of the casting trials indicated that electrolyte additions of 0.05 percent sodium carbonate and 0.10 percent sodium silicate (“N” Brand) were suitable for all body compositions. Approximately 10 gal of slip were prepared from each body composition with this addition of electrolyte. The slips were adjusted to 1.80 specific gravity and de-aired in a vacuum mixer.

*d Details of procedures used in conducting the initial casting trials may be found in the Appendix.
Test specimens were formed by casting 7 in. lengths of $\frac{3}{4}$ in. diam rods in plaster molds. Sets of 15 specimens were fired to six temperatures in an electrically-heated kiln at a firing rate of 75 deg F per hr to 2000 deg F and 50 deg F thereafter until the desired temperatures were reached; a 2-hr soak was maintained at the peak temperatures. No cooling control was attempted.

Results

The results of casting trials made with Series I sanitary ware bodies given in Table 12 show that Custer and Buckingham feldspars may be used interchangeably with very little change in the casting characteristics of the body slip. The use of F-4 feldspar as a replacement for a potash feldspar would cause slightly more rapid casting rates at an equivalent electrolyte level. The best casting slips of the series were found to be those containing A-3 feldspar.

Figure 12 presents the results obtained from the physical tests on fired specimens of sanitary ware bodies. In Series I, the data show that the body...
containing F-4 feldspar achieved maturity in the range of cone 9° to 9°. Bodies containing Custer or Buckingham feldspar matured at a slightly higher temperature, between cone 9° and cone 10°. The body containing A-3 feldspar was found to mature in the range of cone 10° to cone 10°. The maximum moduli of rupture in this series were found at temperatures slightly above the point at which zero porosity was indicated.

The tests indicated that more shrinkage may be anticipated with F-4 feldspar than with equivalent amounts of high potash feldspars such as Buckingham or Custer. The ultimate transverse strengths of the potash feldspar bodies were somewhat greater than those obtained with F-4 feldspar bodies, although F-4 feldspar bodies showed higher strengths at equivalent firing temperatures up to the point of overfiring. It should be noted, however, that at equivalent firing temperatures the porosity of the F-4 feldspar body was lower.

Results obtained with Series II and Series III bodies illustrated the effective fluxing action induced by small additions of tremolitic talc. In compositions containing 32 percent A-3 feldspar, a 2 percent talc addition reduced the maturing temperature range from cone 10°-10° to cone 9°-9°, while a 4 percent addition of talc effected maturity in the range of cone 7°-8°. In a like manner, 2 percent talc, added to similar bodies containing Buckingham or Custer feldspars, reduced the maturing ranges from cone 9°-10° to cone 8°-9°, and 4 percent talc caused maturity at cone 7°-8°. The maturing ranges of F-4 feldspar bodies were reduced from cone 9°-9° to cone 8°-9° and cone 7°-7° by additions of 2 and 4 percent tremolitic talc. The addition of auxiliary flux material apparently had little effect on the ultimate transverse strength of the bodies investigated but caused a slight increase in shrinkage.

Bodies of reduced feldspar content (reduced from 32 to 28 percent) indicated that the 4 percent reduction raised the maturing temperature by slightly more than one cone. A 2 percent talc addition to bodies containing 28 percent feldspar affected maturity in about the same temperature range as those bodies containing 32 percent feldspar with no auxiliary flux additions. The reduced amount of feldspar was observed to yield bodies of slightly lower ultimate strength but of similar shrinkage.

18. Floor Tile

Composition

Four bodies of a representative floor tile composition were prepared using the four commercial feldspars A-3, Buckingham, Custer and F-4 as total flux contents. The specific compositions are shown in Table 13.

<table>
<thead>
<tr>
<th>Body No.</th>
<th>A-3 Feldspar</th>
<th>Buckingham Feldspar</th>
<th>Custer Feldspar</th>
<th>F-4 Feldspar</th>
<th>Victoria Ball Clay</th>
<th>Kamek Kaolin</th>
<th>E.P.K. Florida Kaolin</th>
<th>Tremolitic Talc No. 1</th>
<th>Ottawa Flint</th>
</tr>
</thead>
<tbody>
<tr>
<td>FT1A</td>
<td>55</td>
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<td></td>
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<tr>
<td>FT1B</td>
<td>55</td>
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<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>FT1C</td>
<td>55</td>
<td></td>
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<td></td>
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<td></td>
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<tr>
<td>FT1F</td>
<td>55</td>
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</tbody>
</table>

Processing and Firing

The floor tile bodies were prepared by dry mixing with 8 percent of water. The mixed materials were aged for 24 hr and granulated through a 10-mesh screen, after which specimens were formed by pressing at 2000 psi. Specimens consisted of 1 in. square bars 6 in. in length. Firings were made in an electric kiln at a temperature rate of 100 deg F per hr to 2120, 2150, 2170, 2200, and 2230 deg F. These temperatures represented pyrometric cone equivalents of 4°, 5°, 6°, 7°, and 8°.

Results

The results obtained from tests on the floor tile bodies are shown in Fig. 13. The data show a rapid drop in the porosities of all bodies in the temperature range of cone 4 to cone 8. The body containing F-4 feldspar reached vitrification at a temperature slightly above cone 7°. The potash feldspar bodies achieved vitrification at cone 8°, while the A-3 feldspar body was still incompletely vitrified at that temperature.

The shrinkages obtained with all bodies were approximately of the same magnitude; this was also observed to be the case with the modulus of rupture values.
Fig. 13. Fired Properties of Floor Tile Bodies
IV. SUMMARY OF RESULTS

The results of this extensive study on the use of high potash and intermediate alkali feldspars in a wide range of whiteware bodies may be summarized as follows:

1. Intermediate alkali feldspars may be used interchangeably with high potash feldspars in all types of whiteware bodies with minor changes in the firing characteristics and properties.

2. In whiteware bodies of low feldspar content, such as semi-vitreous bodies, intermediate alkali feldspars with low free quartz content may be interchanged with high potash type feldspars with little or no change in the firing characteristics or fired properties if no other fluxes are present.

3. In whiteware bodies of moderate and high feldspar contents, such as electrical porcelain, sanitary ware and floor tile, an intermediate alkali feldspar such as F-4 may be substituted for potash feldspars to effect maturity at a lower temperature with only a slight reduction in strength and a small increase in shrinkage.

4. Feldspars of high potash content may be used interchangeably at all levels with small differences in the firing characteristics of bodies.

5. Whiting and talc are beneficial in reducing the maturing temperatures of whiteware bodies when added in amounts of 2 to 4 percent.

6. Whiting is a more active flux in bodies containing high potash feldspars than in similar bodies containing intermediate alkali feldspars.

7. Talc is a more active flux in bodies containing intermediate alkali feldspars than in similar bodies made with high potash feldspars.

8. The over-all fluxing action of talc was found to be more effective than equivalent amounts of whiting in bodies containing either type of feldspar.

9. Magnesite was found to be a powerful flux when added in amounts of 2 percent, but caused appreciable increases in the fired shrinkage.

10. The addition of auxiliary fluxes was found to shorten the firing range of bodies in which they were used.

11. Considerable cristobalite formation was observed in semi-vitreous bodies containing intermediate alkali feldspars and MgO-bearing fluxes such as talc and magnesite. Little or no cristobalite was found in similar bodies made with high potash feldspars.

12. Some cristobalite formation was found in all whiteware bodies containing intermediate alkali-feldspars.

13. X-ray studies revealed that the formation of cristobalite took place during the cooling cycle with crystal deposition of SiO$_2$ or devitrification.
APPENDIX

19. Raw Materials Used in Experimental Bodies

The chemical analyses of the feldspars used in the experimental bodies are shown in Table 14. The analyses of other materials used in making various body compositions are also given in Table 14.

20. Details of Body Preparation

Blunging

Blunging was used for the blending of raw materials in preparation of semi-vitreous, electrical porcelain and sanitary ware bodies. The blunging equipment consisted of a 20-gal tank with two sets of paddles which were rotated in opposite directions to create turbulent counterflow blunging. Approximately 12 gal of water was used for the blunging of bodies; 120 lb of dry weight of each body composition was slowly added to the water with the paddles rotating. The materials of each batch were added in the order of decreasing plasticity; the ball clays were added first, followed by the kaolins and then the non-plastic materials. Sufficient time was allotted between the various additions to allow the slip to become smooth. After all materials had been added, the slip was allowed to blunge for approximately 1 hr and then passed through a 120-mesh lawn and over a set of magnets to remove any particles of iron. The processed slip was stored in smooth blungers for filter pressing.

Ball Milling

Ball milling was used to prepare the hotel china bodies. A 35-gal Patterson direct motor-driven ball mill was used. The mill charge consisted of 120 lb of dry batch and 10 gal of water. Each batch was milled for a period of 4 hr after which it was passed through a 120-mesh lawn and over magnets. The ball-milled batches were placed in smooth blungers for storage until ready for filter pressing.

Dry Mixing

Dry mixing was employed for the blending of raw materials in the preparation of floor tile bodies. Twenty-five lb of dry batch was added to a No. 0 Simpson Muller-mixer and allowed to mix for 5 min, after which 1 qt of water was added and mixed an additional 15 min. The water addition made was approximately 8 percent of the dry weight of the body. The mixed material was placed in sealed jars and aged for 24 hr, after which it was granulated through a 10-mesh screen and replaced in the storage jars until ready for use in pressing.

Filter Pressing and Pugging

All of the bodies prepared by blunging or ball milling were filter pressed and subsequently pugged. The processed slips of each body were pumped from the smooth blungers and filter pressed to a pressure of 140 psi for 45 min; the resultant filter cakes contained approximately 20 percent of water. The filter cakes of each body composition were pugged into slugs 3 in. in diam by 12 in. long and placed in sealed damp jars for one week of aging. The pugging operation was used to assure a uniform moisture distribution within each body and also to facilitate the storage during the aging period.

21. Specimen Formation

Extrusion

Specimens for the testing of semi-vitreous, electrical porcelain and hotel china bodies were prepared by vacuum extrusion with an International Vac-aire De-airing Extrusion Machine. Specimens were extruded as round rods 7 in. in diam; then 80 rods cut 7 in. in length and 40 rods cut 3½ in. in length for each body composition. The specimens were allowed to air dry in V-shaped troughs and then placed in an oven at 220 deg F to complete the drying.

Casting

Test specimens of sanitary ware bodies were made by casting in plaster of Paris molds. Previous to the preparation of the casting slips of each body, six small slip batches were prepared from each body composition to determine the casting characteristics. Each trial slip batch was prepared from slugs which had been aged for one week. The water content of each body slug was determined by the difference in weight before and after thorough dry-
ing of 100 grams of plastic slug. The percent moisture content based on the weight of dry solids was obtained as follows:

$$\% \text{ Moisture (dry basis)} = \frac{\text{Plastic Weight} - \text{Dry Weight}}{\text{Dry Weight}} \times 100$$

The weight of plastic material equivalent to 100 grams of dry body was calculated as follows:

$$1000 \left(1 + \frac{\% \text{ Moisture}}{100}\right) \text{grams}$$

It was desired to prepare slips with a specific gravity of approximately 1.80; by experiment it was found that when the solid to water ratio was approximately 72 to 28 a specific gravity of slightly more than 1.80 was obtained. Slips were prepared with this solid to water ratio. The electrolyte contents of the trial slips were based on the dry weight of solids and were as follows:

<table>
<thead>
<tr>
<th>Percent Sodium Carbonate</th>
<th>Sodium Silicate (&quot;N&quot; Brand)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. 0.05</td>
<td>+ 0.05</td>
</tr>
<tr>
<td>2. 0.05</td>
<td>+ 0.075</td>
</tr>
<tr>
<td>3. 0.05</td>
<td>+ 0.100</td>
</tr>
<tr>
<td>4. 0.05</td>
<td>+ 0.125</td>
</tr>
<tr>
<td>5. 0.05</td>
<td>+ 0.150</td>
</tr>
<tr>
<td>6. 0.05</td>
<td>+ 0.175</td>
</tr>
</tbody>
</table>

The electrolytes were added to the water required for each body slip and thoroughly dispersed before adding any of the plastic body. A small motor-driven propeller type mixer was used for mixing the plastic body into the electrolyte solution. The trial slips were mixed to a smooth consistency, passed through a 120-mesh screen and adjusted to a specific gravity of 1.80. The slips were then transferred to Mason jars which were placed in a vacuum chamber for de-airing of the slip. After de-airing, the jars of slip were set in a tumbling apparatus and slowly tumbled end over end for 48 hr.

At the end of the tumbling period, the specific gravity of each slip was determined by weighing 100 cc of slip. The flow properties of each were determined by the time required for 100 cc of slip to flow through a \(\frac{3}{4}\) in. diam orifice of a Mariotte flow tube. Three determinations were made with each slip sample.

The relative casting rates of the various slips were determined by casting two 1 in. square bars and allowing each slip to “set up” for 40 min after which the molds were inverted and allowed to drain for 5 min. The molds were opened after the end of the draining period and the cast specimens removed; the nature of drainage and type of cast was noted. A 7 in. length of each cast bar was cut and immediately weighed to obtain the wet cast weight. The cast specimens were dried completely and reweighed to obtain the dry cast weight. The percent water retention of the cast specimens was calculated as follows:

$$\% \text{ Water Retention} = \frac{\text{Wet Cast Weight} - \text{Dry Cast Weight}}{\text{Wet Cast Weight}} \times 100$$

The molds used for the casting trials and all other casting were prepared from a mixture of 65 percent gypsum plaster and 35 percent water.

From the casting trials described above, it was found that an electrolyte addition of 0.05 percent sodium carbonate + 0.10 percent of “N” Brand sodium silicate, based on the dry weight of the body, was suitable for the casting of all body compositions. Using this electrolyte content, the plastic bodies were made into slips of 1.80 specific gravity.

<table>
<thead>
<tr>
<th>Chemical Analyses of Raw Materials Used in Experimental Bodies</th>
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<tbody>
<tr>
<td>SiO₂</td>
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<tr>
<td>----------------------</td>
</tr>
<tr>
<td>Tremolitic</td>
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<tr>
<td>Sierramic Low</td>
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<tr>
<td>Lime Tate</td>
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<tr>
<td>Wash, Dead</td>
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<tr>
<td>Burned Magnesite</td>
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<td>E.P.K. Fla. Kaolin</td>
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<tr>
<td>Pioneer</td>
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<tr>
<td>Tenn. No. 5</td>
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<tr>
<td>Ball Clay</td>
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<tr>
<td>Old Mine No. 4</td>
</tr>
<tr>
<td>Ball Clay</td>
</tr>
<tr>
<td>Victoria</td>
</tr>
<tr>
<td>Imperial</td>
</tr>
<tr>
<td>Royal Ball</td>
</tr>
<tr>
<td>Ottawa</td>
</tr>
</tbody>
</table>

* W. H. Loomis Tate Corp., New York, N.Y.
† Sierra Tate Co., Los Angeles, Calif.
¶ Edgar Plastic Kaolin Co., Middletown, N.J.
§ Harris Clay Co., Spruce Pine, N.C.
¶¶ Georgia Kaolin Co., Elizabeth, N.J.
†† Kentucky Tennessee Clay Co., Mayfield, Ky.
**** United Clay Mines Corp., Trenton, N.J.
***** Ottawa Silica Co., Ottawa, Ill.
using the same type calculations as described for the casting trials. The slips were mixed for 4 hr and allowed to age in stoneware jars for 24 hr, after which they were remixed for 30 min and passed through a 120-mesh lawn. De-airing of the slips was accomplished in a vacuum mixer.

Specimens were cast into rods ½ in. in diam and 7 in. in length. Plaster of Paris molds were used and the specimens were allowed to cast solid. Upon removal from the molds, eighty 7 in. lengths and forty 3½ in. lengths were cut and allowed to air dry for 3 days; drying was completed at 200 deg F in an oven dryer.

**Dry Pressing**

Floor tile specimens were prepared by dry pressing using a Denison “Hydroilic” press. Seventy-five bars 1 in. square by 6 in. long were pressed from each body composition at a pressure of 2000 psi.

### 22. Test Procedures

#### Volume Shrinkage

The firing volume shrinkages of all bodies except those of floor tile were obtained with the 3¼ in. length specimens. Prior to firing, the dry volumes of these specimens were obtained from the saturated and suspended weights in kerosene. Each piece was placed in kerosene until saturated; dry volume was calculated as shown below:

\[
\text{Dry Volume} = \frac{\text{Saturated Weight} - \text{Suspended Weight}}{\text{Specific Gravity of Kerosene}}
\]

After firing, the fired volumes were obtained in a similar manner using the saturated and suspended weights in water. The volume shrinkage was determined as follows:

Percent Volume Shrinkage = \( \frac{\text{Dry Volume} - \text{Fired Volume}}{\text{Dry Volume}} \times 100 \)

The reported values of volume shrinkage are the average of five specimens.

The volume shrinkages of floor tile bodies were calculated from linear shrinkage determinations. Linear shrinkage was calculated from the equation:

Percent Linear Shrinkage = \( \frac{\text{Dry Length} - \text{Fired Length}}{\text{Dry Length}} \times 100 \)

The conversion from linear to volume shrinkage is determined as follows:

\[
\text{Percent Volume Shrinkage} = 100 \left(\frac{a}{100} - 1\right)^3 - 1
\]

where \( a \) equals the percent linear shrinkage.

#### Fired Modulus of Rupture

The modulus of rupture values reported were obtained as the average of a minimum of 12 specimens. Specimens were stored in dessicators immediately after firing to prevent any moisture absorption. A Dillon Dynamometer with a 5 in. span between knife edges was employed for the determination of the cross-breaking load.

If \( P \) is the cross-breaking load in lbs, \( l \) is the span between knife edges in in. and \( d \) is the diam of a round specimen at the point of fracture; then the modulus of rupture \( (M) \) is calculated as:

\[
M = \frac{8P l}{d^3 \pi}
\]

(for specimens of round cross section)

For square bars, such as the floor tile specimens,

\[
M = \frac{3P l}{2b d^2 \pi}
\]

(where \( b \) and \( d \) are the breadth and depth of the square bar at the point of fracture)

#### Fired Porosity

The fired porosities were determined on the fractured portions of modulus of rupture specimens. The fractured portions were thoroughly dried in an oven at 220 deg F and allowed to cool to room temperature in a dessicator. The weights of the dry pieces were determined after which the pieces were placed in water which was brought to a boil and allowed to boil for 8 hr. After cooling to room temperature, the specimens were removed from the water, wiped with a damp cloth to remove any excess moisture and weighed. The suspended weights of the saturated pieces were also determined. The porosity was calculated as follows:

Percent Porosity = \( \frac{\text{Fired Saturated Weight} - \text{Fired Dry Weight}}{\text{Fired Saturated Weight} - \text{Fired Suspended Weight}} \times 100 \)

#### X-Ray Analysis

The powder diffraction method was used for qualitative and quantitative analyses of the crystalline content of fired specimens. This study required a Norelco Geiger-Counter X-ray Spectrometer with automatic recorder.
The method of sample mount was that proposed by McCreery. The sample holder consisted of an alloyed aluminum cell, 1½ in. x 1 in. x ½ in. with a 3/16 in. hole at the center.

Samples for X-ray analysis were obtained by cutting a 1/4 in. x 1/2 in. section from the centers of specimens. These segments were crushed and ground in an agate mortar to pass a 325-mesh screen. Iron impurities were removed by passing a permanent magnet through the finely ground material. Samples designated for quantitative analysis were thoroughly dried after grinding and 1.000 gram of each was mixed with chemically-precipitated calcium fluoride in an agate mortar with 5 ml of methyl alcohol. The alcohol was removed by evaporation and the samples were mixed dry for an additional 10 min.

Standard samples for the determination of calibration curves were prepared from Ottawa flint, electric furnace mullite, prepared cristobalite and calcium fluoride with fused F-4 feldspar glass as a diluent. Considerable difficulty was encountered in analyzing the mullite content because of its tendency toward preferred orientation.

Quantitative analyses were made according to methods described by Tuttle and Cook in which the ratio of the height of a peak to the height of the internal standard peak (calcium fluoride) was compared to the calibration ratio curve obtained with standard mixtures. Peak intensities were also determined by the counting method and also by measurement of peak heights on the recorder chart operating at 1° (2 theta) per min. Using the chart method, three runs for each of three mixings of each sample were made.

The major quartz and mullite peaks at $d = 3.35$ and 3.43 respectively could not be used due to their juxtaposition and the tendency of each to reinforce the other. In most cases the mullite peak could not be separated from the more intense quartz peak. The peaks used in the analyses were those found at $d$-values of 4.24 for quartz, 4.05 for cristobalite and 2.70 for mullite.

Both copper and iron radiation were used for the X-ray analyses, but iron radiation was found to give more resolution and also shifted the peak locations to higher angles on the goniometer arc where the background interferences were lower.
BIBLIOGRAPHY


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