TWO NEW CRYSTALLINE PHASES OF 
THE ANORTHITE COMPOSITION, 
CaO · Al₂O₃ · 2SiO₂

G. L. DAVIS AND O. F. TUTTLE

ABSTRACT. Two new crystalline phases of the composition CaAl₂Si₂O₈ are described, giving the optical properties and X-ray powder diffraction data. Their synthesis and stability are discussed. Comparisons are drawn with similar modifications of BaAl₂Si₂O₈.

INTRODUCTION

DURING the investigation of subsolidus phase relations in the system: albite—anorthite, an unknown phase appeared at temperatures near 1000° C. in compositions rich in lime feldspar. The unknown phase was found to be identical in optical and X-ray properties with one of the two principal phases of unknown composition crystallized by L. B. Wyckoff of the Research Laboratory of the Electrometallurgical Company of the Union Carbide and Carbon Corporation. This material, which was submitted by Wyckoff to L. H. Adams of the Geophysical Laboratory for examination, was prepared by fusing a large charge of several hundred grams of glass of the approximate composition of anorthite (CaO·Al₂O₃·2SiO₂) at 2000° C. in a graphite crucible in a furnace with a nitrogen atmosphere. The melt was cooled slowly to 1250° and the charge was allowed to crystallize at this temperature. Crystallization resulted in segregation of the two crystalline phases in such a manner that it was possible to hand-pick the two for chemical analysis.

The two new phases will be called hexagonal (Donnay, 1952) and orthorhombic¹ CaAl₂Si₂O₈.

CHEMICAL COMPOSITION

The two phases crystallized by Wyckoff were separated by hand-picking under a binocular microscope and analyzed chemically by conventional methods. The results, with the theoretical composition of anorthite for comparison, were:

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¹ Laves, F., personal communication.
These analyses are the averages of duplicate determinations on 100 mg. samples. The composition is therefore essentially that of anorthite, CaO·Al₂O₃·2SiO₂.

**GENERAL CHARACTER**

The hexagonal phase crystallizes as spherulitelike masses consisting of transparent radiating basal plates. A perfect basal cleavage yields cleavage plates which are brittle and break with a conchoidal fracture. No evidence of twinning has been observed. It appears to have a hardness of 5 on the cleavage surface but a polished surface across the cleavage has a hardness of 6. The density determined on a 5 mg. fragment is 2.74.

The orthorhombic phase crystallizes as a massive mosaic of transparent equant crystals which show no evidence of crystal faces. Crushed fragments show a conchoidal fracture. The hardness is 6 and the density 2.70. This form has poor cleavage parallel to the optic plane. There are numerous inclusions aligned in planes parallel to the optic plane.

**OPTICAL PROPERTIES**

The optical properties of the two new phases are given together with those of anorthite for comparison:

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**X-RAY DATA**

Powder diffraction records were made on the Philips Wide-range Spectrometer. The values obtained are given in tables 1 and 2, which also present the results obtained by applying the method of indexing powder records proposed by C. Runge (1917) and recently generalized by T. Ito (1950). The radiation employed was CuKα with a₁ resolved at all angles above 32° (2θ). A .0007-inch nickel filter was used. In the tables, intensities are expressed relative to a strong line, not necessarily the strongest, and Q is the square of the reciprocal lattice
Phases of the Composition CaO·Al₂O₃·2 SiO₂  109

vector. The character of the powder pattern for the hexagonal phase indicates that the material examined was preferentially oriented.

The hexagonal material has the following lattice parameters: \( a = 5.110 \pm .002 \ \text{Å}, \ c = 2 \times 7.367 \pm .002 \ \text{Å}, Z = 1 \). The calculated density is 2.77, which compares with 2.74 measured. This material has been studied by Donnay (1952) and assigned the space group \( C \ 6/mmm \).

The orthorhombic phase has the unit cell dimensions \( a = 8.224 \pm .006 \ \text{Å}, b = 8.606 \pm .006 \ \text{Å}, c = 4.836 \pm .005 \ \text{Å}, \) and \( Z = 2 \). The only systematic absences are for reflections with \((h + k)\) odd in \((hk0)\). A. J. Frueh, Jr., of the University of Chicago, has pointed out in a personal communication that

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etc.

\( a = 5.110 \pm .002 \ \text{Å}, \ c = 2 \times 7.369 \pm .002 \ \text{Å}, Z = 1 \)

* Specimen shows preferred orientation.
### Table 2
Orthorhombic Form of CaO·Al₂O₃·2 SiO₂

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\[ a = 8.224 \pm 0.006 \text{ Å}, \quad b = 8.606 \pm 0.006 \text{ Å}, \quad c = 4.836 \pm 0.005 \text{ Å}, \quad Z = 2 \]
the space group is $P_{\text{1}2_{1}2_{1}}$ and that the absence (h + k) odd in (hk0) is only a pseudo-absence. These reflections are too weak to be observed in a powder pattern. The calculated density is 2.70 which agrees with the measured value.

**LABORATORY INVESTIGATION**

Orthorhombic CaAl$_2$Si$_2$O$_8$ apparently can be synthesized, in the absence of water vapor, only by crystallizing the glass at temperatures well below the liquidus. The largest yields have been obtained at temperatures just above the temperature at which the glass fails to crystallize. It was first observed in material obtained by crystallizing a glass of composition Ab$_{20}$An$_{80}$ at 1000° C. At 950° C. the glass crystallized essentially completely as the orthorhombic phase in four days. Glass of anorthite composition at 950° for four days gave some of the orthorhombic phase together with anorthite and the hexagonal modification. This glass failed to crystallize in four days at 890° C. By these methods a large yield of the orthorhombic form has never been obtained from anorthite glass. On the other hand, glasses having compositions in the range Ab$_{10}$An$_{90}$ to Ab$_{70}$An$_{30}$ crystallized predominantly as the orthorhombic form when held from two to four days between 950° and 1000° C. A small amount of the orthorhombic phase was detected in the poorly crystallized product when glass of composition Ab$_{80}$An$_{20}$ was heated at 1000° C. for twenty-one days.

The orthorhombic material crystallized from glasses rich in soda has noticeably lower refractive indices than the same phase crystallized from glass of anorthite composition, indicating considerable solid solution of NaAlSi$_3$O$_8$. The X-ray powder records confirm the existence of this solid solution which is shown by the change in the difference between 2θ (002) and 2θ (021) with increasing NaAlSi$_3$O$_8$.

Hexagonal CaAl$_2$Si$_2$O$_8$ has been synthesized only as a minor component of the crystalline product in compositions ranging from Ab$_{80}$An$_{20}$ to anorthite and its presence has been determined by X-ray powder patterns solely. It has not been identified optically. As in the case of the orthorhombic form, there is considerable solid solution with NaAlSi$_3$O$_8$ indicated by changes in the (004) spacing with increasing NaAlSi$_3$O$_8$.

A large yield of this phase has been obtained only by
repeating the conditions under which Wyckoff synthesized the materials in the course of his viscosity studies. A glass of anorthite composition heated to 2000° C. in a graphite resistor trough in a vacuum fusion furnace (Evans, 1935; Urry and Piggot, 1941) and quickly cooled to approximately 1200°, where it was allowed to crystallize, gave the hexagonal phase only. In this treatment there it some evidence that some of the silica is reduced. Possibly the slight compositional change may promote the growth of this phase.

COMPARISON WITH SIMILAR MODIFICATIONS OF BaAl$_2$Si$_2$O$_8$

Synthetic hexagonal BaAl$_2$Si$_2$O$_8$ has been reported by Ito (1950), and a structure has been proposed for this material. It was synthesized in an environment similar to that used for producing hexagonal CaAl$_2$Si$_2$O$_8$. An inversion of the high-low type has been reported for the hexagonal BaAl$_2$Si$_2$O$_8$ at 300° C. (Yoshiki and Matsumoto, 1951). We have confirmed the existence of this inversion. Differential heating experiments show an absorption of heat beginning at 295° ± 5° C. Differential heating measurements on hexagonal CaAl$_2$Si$_2$O$_8$ failed to detect a heat effect up to 1200° C.

We have synthesized hexagonal BaAl$_2$Si$_2$O$_8$ by heating the oxides at 1500° C. for four days. However, longer heating caused it to change to celsian (monoclinic feldspar, BaAl$_2$Si$_2$O$_8$), a behavior like that of the hexagonal lime compound. Donnay (1952) reports that hexagonal CaAl$_2$Si$_2$O$_8$ is not isostructural with the barium compound, as described by Ito. However, hexagonal CaAl$_2$Si$_2$O$_8$ may be isostructural with the high temperature form of hexagonal BaAl$_2$Si$_2$O$_8$.

Paracelsian (BaAl$_2$Si$_2$O$_8$), described by L. J. Spencer (1942) is orthorhombic morphologically, and the possibility that this material is isostructural with orthorhombic CaAl$_2$Si$_2$O$_8$ was considered. More recently, Smith (1952) has found paracelsian to be monoclinic, departing only slightly from orthorhombic symmetry. There is no relation between the structure of paracelsian and the orthorhombic form of CaAl$_2$Si$_2$O$_8$ since they have completely different space groups, that of paracelsian being the pseudo space group $Pbnm$ or $Pbn$. Paracelsian changes readily to celsian on heating, being similar in this respect to the orthorhombic lime compound.
STABILITY OF THE HEXAGONAL AND ORTHORHOMBIC CaAl₂Si₂O₈

Both the hexagonal and the orthorhombic forms change to anorthite on prolonged heating at the temperature at which they have been synthesized. The change takes place rapidly at high temperatures and becomes more difficult to produce as the temperature is lowered. However, in the presence of water vapor at 1000 kg/cm², both forms invert to anorthite at temperatures well below (700° C.) those at which no change can be detected when heated in the absence of water. Their possible stability at lower temperatures is being investigated by J. R. Goldsmith and E. G. Ehlers² of the University of Chicago.

The fact that these new phases have not been found in nature, in addition to the present lack of evidence for a stable temperature range of existence in the laboratory, suggests that they are unstable. High temperature modifications of silicates usually have higher symmetry than the low temperature forms. The symmetry relations of the three modifications of anorthite composition would suggest that the orthorhombic and hexagonal forms should be high temperature modifications but the rapid change of both phases to anorthite at high temperature appears to eliminate this possibility.

If further experiments at low temperature in the presence of fluxes fail to indicate a stable temperature range for the orthorhombic and hexagonal forms of CaAl₂Si₂O₈, it must be concluded that both are unstable modifications.

Laboratory studies of silicates crystallized from viscous glasses have repeatedly demonstrated that this environment promotes the formation of metastable and unstable phases. It is not surprising, then, that these unusual forms have been encountered in our studies of subsolidus relations of the feldspars. It is anticipated that other unstable phases will be discovered as our studies of subsolidus relations are extended to other silicate systems.

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² Personal communication.
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