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STUDIES IN THE SYSTEM BaO–Al_2O_3–SiO_2
II. THE BINARY SYSTEM CELSIAN ($BaAl_2Si_2O_8$)–SILICA (SiO_2)

W. R. FOSTER and H. C. LIN*

Department of Mineralogy, Ohio State University, Columbus, Ohio 43210

ABSTRACT. The results of quenching experiments on selected mixtures on the $BaAl_2Si_2O_8$–SiO_2 join of the BaO–Al_2O_3–SiO_2 system are presented. A simple eutectic system is shown to exist between the two end-members, the eutectic point being located at 1311 ± 4°C, and at a composition of 51 percent $BaAl_2Si_2O_8$ and 49 percent SiO_2. The complex relations between celsian and silica reported in earlier investigations have not been confirmed. Unlike the latter, the present results are in full accord with the coexistence of celsian and quartz in natural mineral occurrences.

INTRODUCTION

Because of their great petrological and technological significance, ternary systems of alumina and silica with the alkalis and the alkaline earths have been under continuing investigation by experimental mineralogists. Interest in the system BaO–Al_2O_3–SiO_2 has centered largely on the compound $BaAl_2Si_2O_8$. In spite of its limited occurrence in nature this compound has noteworthy affiliations with the geologically important feldspar group of minerals. The great majority of feldspars contain small quantities of barium (Deer, Howie, and Zussman, 1963), which is considered to be present in solid solution as $BaAl_2Si_2O_8$. Occasionally the $BaAl_2Si_2O_8$ content is substantial (10 to 30 percent), forming hyalophane and barium plagioclase. These may be regarded as intermediate members of the $KAlSi_3O_8$–$BaAl_2Si_2O_8$ and $CaAl_2Si_2O_8$–$BaAl_2Si_2O_8$ series, respectively. The relatively rare plagioclases in which $BaAl_2Si_2O_8$ predominates over $CaAl_2Si_2O_8$ are referred to as calcioclesian and celsian, the latter containing 90 percent or more of $BaAl_2Si_2O_8$.

The thermal behavior and phase relations of celsian are less completely understood than those of the more common feldspar minerals. Its polymorphic transformations have recently been studied, with widely varying results, by Sorrell (1962), Seki and Kennedy (1964), and Lin and Foster (1968), among others. Roy (1965) has conducted a hydrothermal investigation of the $KAlSi_3O_8$–$BaAl_2Si_2O_8$ system, but no recent experimental study appears to have been made of the $CaAl_2Si_2O_8$–$BaAl_2Si_2O_8$ system. Although substantial solid solution exists in both of these series, there is as yet no conclusive evidence that the solid solution is complete, at least in the natural series. No study has yet been made of the ternary systems involving each of these feldspar series with free silica. Thomas (1950) and Toropov, Galakhov, and Bondar (1954) have examined the phase relations of $BaAl_2Si_2O_8$ with other phases in the system BaO–Al_2O_3–SiO_2. Certain aspects of the latter two studies are at odds with the relations that might be expected on the basis of natural mineral assemblages. Accordingly, a series of investigations in-

* Present address: Department of Geology, Queen's University, Kingston, Ontario, Canada

volving celsian in the presence of sanbornite ($BaO.2SiO_2$), the silica minerals (SiO_2), mullite ($3Al_2O_3.2SiO_2$), and corundum (Al_2O_3), has been undertaken. The present paper reports only on the study of the celsian-silica system.

REVIEW OF PREVIOUS WORK

The system celsian ($BaAl_2Si_2O_8$)-silica (SiO_2) has not previously been studied as a discrete system, nor has a phase diagram been presented. However, results of investigations of the general ternary system BaO–Al_2O_3–SiO_2 (Thomas, 1950; Toropov, Galakhov, and Bondar, 1954) permit the deduction of equilibrium relations for the celsian-silica series of compositions. Figure 1 depicts such relations, as derived by the present authors from the ternary diagrams (figs. 2 and 3) of the two cited studies. Both investigations indicated celsian to be incompatible with silica where studied, there being no conditions of temperature and composition under which these two phases coexist. In the liquidus range of temperatures of figure 1 the liquidus curve of mullite intervenes between the liquidus curves of celsian and silica. Such a relation, in itself, does not preclude the coexistence of celsian and silica at the solidus, through a reaction relation whereby mullite is resorbed by the liquid. But figure 1 is unequivocal in excluding the formation of a celsian-silica assemblage by such a reaction relation, indicating instead the solid phase assemblages celsian–sanbornite–mullite, sanbornite–mullite, and silica–sanbornite–mullite.

There are several reasons for skepticism about the phase relations depicted in figure 1. The natural occurrence, with quartz, of barium feldspar approximating celsian in composition (Schaller, 1929; Rogers, 1932; Segnit, 1946) is at least suggestive of the coexistence of celsian and silica in the experimental system. Experimental studies in the appropriate ternary systems have already confirmed the compatibility with silica of calcium feldspar (Rankin and Wright, 1915), of strontium feldspar (Dear, 1957), of potassium feldspar (Schairer and Bowen, 1955), and of sodium feldspar (Schairer and Bowen, 1956). It might reasonably be expected, then, that barium feldspar would display analogous relations in the BaO–Al_2O_3–SiO_2 system. The results of the hydrothermal studies of Seki and Kennedy (1964) establish the coexistence of celsian and quartz at least as high as 800°C. In preliminary experiments by the present authors, an appropriate mixture of sanbornite ($BaO.2SiO_2$) and mullite ($3Al_2O_3.2SiO_2$) was converted to celsian and silica upon calcination for 39 hours at 1250°C. In both instances the coexistence of sanbornite and mullite implicit in figure 1 was contradicted. A reinvestigation of the BaO–Al_2O_3–SiO_2 system in general, and of the $BaAl_2Si_2O_8$–SiO_2 system in particular, therefore seemed desirable.

EXPERIMENTAL PROCEDURE

Raw materials used in this study were of chemical reagent grade. C. P. Baker's analyzed barium carbonate of 99.5 percent purity was used as the source of BaO. Alkalies totalled less than 0.07 percent. Sul-

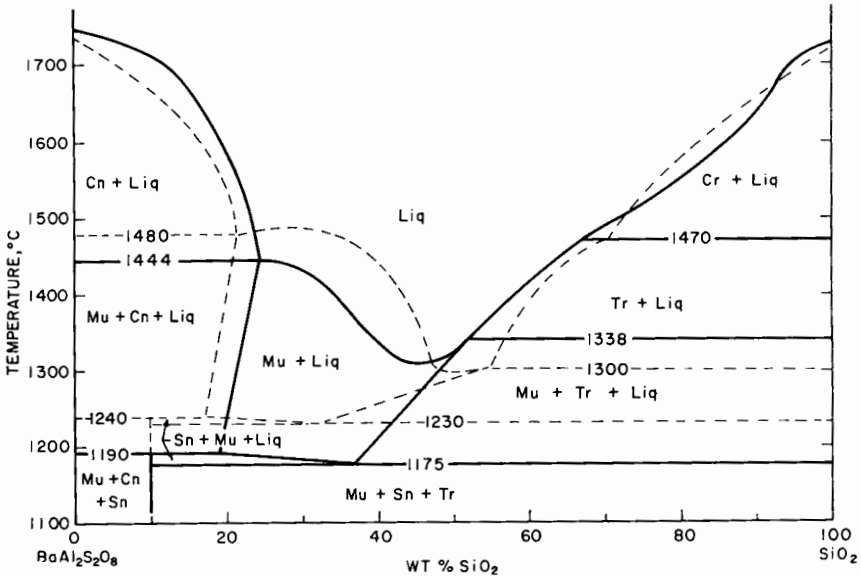

Fig. 1. Phase diagram for the system $BaAl_2Si_2O_8$–SiO_2 as deduced from the results of previous investigators: solid lines, Thomas (1950); dashed lines, Toropov, Galakhov, and Bondar (1954). Symbols: Cn = celsian, Mu = mullite, Sn = sanbornite, Tr = tridymite, Cr = cristobalite, Liq = liquid.

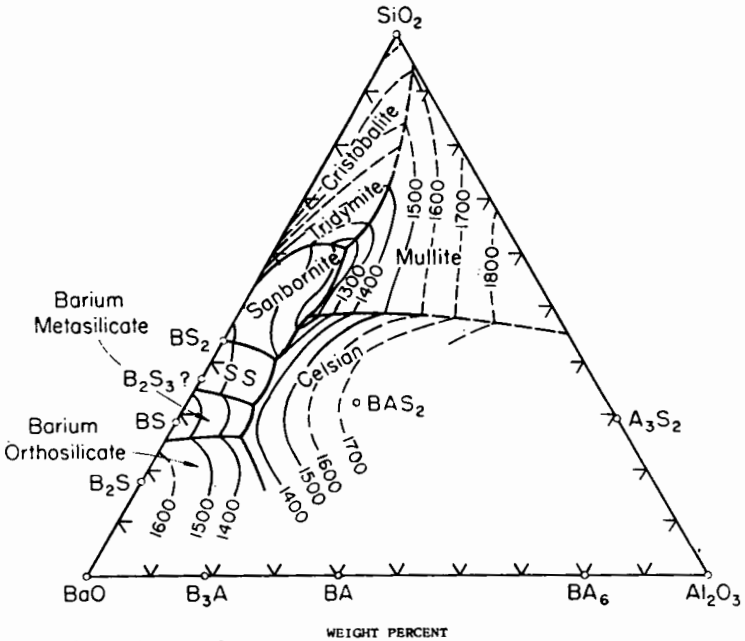

Fig. 2. Phase diagram for the system BaO–Al_2O_3–SiO_2, after Thomas (1950). Symbols: B = BaO, A = Al_2O_3, S = SiO_2, ss = alleged BS_2 solid solution.

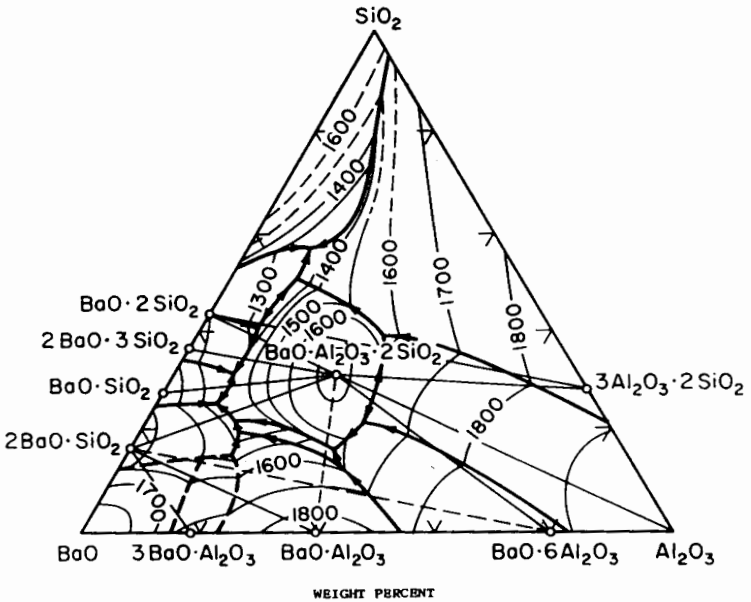

Fig. 3. Phase diagram for the system BaO–Al_2O_3–SiO_2, after Toropov, Galakhov, and Bondar (1954).

fates of calcium and strontium made up the bulk of the remainder. For Al_2O_3, Linde Type B-5125 gamma-alumina of 99.9 percent purity was used. Crystalline quartz in the form of 5-micron "Min-U-Sil" of the Pennsylvania Glass Sand Corporation (purity, 99.9 percent) served as the source of SiO_2.

Homogeneous glasses corresponding to 12 selected compositions between celsian and silica were prepared from the above raw materials. Repeated fusion was carried out in platinum crucibles in the 1300° to 1650°C range until uniformity was obtained. Figure 4 shows the variation of refractive index with composition, as obtained from the present study, as well as from the work of Thomas (1950) and of Toropov, Galakhov, and Bondar (1954).

Glasses so prepared were devitrified as a preliminary step to the phase equilibrium study. Devitrification was effected by heating the glass for 12 to 36 hours between 1200° and 1300°C. This treatment was considered as satisfactory only if both $BaAl_2Si_2O_8$ (as celsian or hexacelsian) and SiO_2 (as cristobalite) were revealed in substantial amounts by X-ray diffractometry. Although interstitial glass was visible microscopically, no crystalline phases other than $BaAl_2Si_2O_8$ or silica were detected. In view of the relations shown by figure 1, it is significant that neither sanbornite nor mullite was identified in any of the devitrified glasses.

Phase relations were established by means of the quenching method developed at the Geophysical Laboratory (Roedder, 1951; Schairer, 1959).

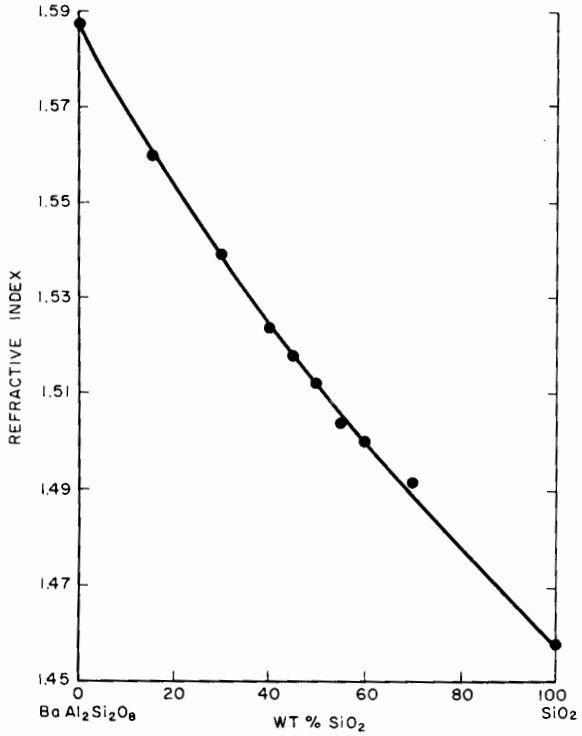

Fig. 4. Refractive index chart for glasses in the $BaAl_2Si_2O_8$–SiO_2 system.

Small charges of devitrified glass were held at selected temperatures in the hot zone of a platinum 60 rhodium 40 resistance furnace for a time sufficient to establish equilibrium. The charges were then quenched in mercury so as to retain the phases present at the elevated temperature. The platinum-platinum 90 rhodium 10 thermocouple used for temperature measurement was calibrated against the melting points of gold (1063°C), diopside (1391.5°C), and gehlenite (1590°C).

Phases were identified with the polarizing microscope, aided in many instances by X-ray powder diffraction patterns. The optical properties which served to distinguish the two forms of $BaAl_2Si_2O_8$ (celsian and hexacelsian) and the cristobalite form of SiO_2 were as follows:

Celsian usually appeared as elongated needles or bladed crystals with inclined extinction up to 28°, although rounded and blocky crystals also occurred. Hexacelsian displayed stubby lath-like crystals with parallel extinction. Cristobalite was readily recognized by its lower refractive index with reference to the surrounding glass and occurred as irregular shred-like patches, globular aggregates, or rounded discrete rains of negligible birefringence. No crystals corresponding in optical properties to tridymite, sanbornite, or mullite were encountered.

Cristobalite invariably appeared in place of tridymite, and hexacelsian often partially substituted for celsian. Such metastable appearance or persistence of one polymorph in the range of temperature stability of another is a common phenomenon in silicate phase equilibrium work. There is no reason to believe that such appearances cast doubt on the essential validity of the phase assemblages or on the general equilibrium relations deduced therefrom.

RESULTS

Approximately 100 runs were made on the 12 different celsian-silica compositions. Only about one-third of these runs were significant for the establishment of phase relations. These are presented in table 1 and have been used to prepare the revised phase diagram for the system, shown in figure 5.

This is a simple binary eutectic system with two congruently melting end-members, celsian ($BaAl_2Si_2O_8$) and silica (SiO_2). The $BaAl_2Si_2O_8$ melting point of 1760°C, as well as the 1590° inversion of celsian to hexacelsian, are taken from the recent study of Lin and Foster (1968). The silica melting point of 1713°C and the 1470°C inversion of tridymite to cristobalite are taken from the work of Fenner (1913). As recently discussed briefly by Rockett and Foster (1967) there is an alternative to Fenner's views on silica transformations, which calls for the elimination of tridymite as a polymorph in the SiO_2-system. The authors know of no convincing evidence for the abandonment of Fenner's interpretation in favor of this alternative.

The liquidus determinations were in most cases established with an accuracy of $\pm$ 3°C or better. Cristobalite was the only phase of silica encountered, even in the stability range of tridymite. Many previous workers have noted such metastable persistence of cristobalite outside its stability range. Retention of tridymite as a polymorph of SiO_2 is based in part on the recent study of Rockett and Foster (1967). The establishment of the silica liquidus curve was made difficult by the great sluggishness with which cristobalite re-dissolves in the devitrified silica-rich glasses, even during prolonged runs. Hexacelsian occasionally accompanied celsian in the temperature range of the latter phase. This is not unexpected, in view of the strong tendency for hexacelsian to form and persist metastably over a wide range of temperatures (Lin and Foster, 1968). No evidence for the existence of a liquidus curve for mullite was found, and neither mullite nor sanbornite was detected, whether optically or by X-ray diffractometry, in the entire temperature range of the investigation. An indication that mullite and sanbornite are incompatible had already been given by the earlier mentioned run at 1250°C for 39 hours.

The eutectic point in the system has been located at a temperature of 1311 $\pm$ 4°C and at a composition of 51 percent celsian and 49 percent silica by weight. The point was fixed by the intersection of the established liquidus curves of $BaAl_2Si_2O_8$ and SiO_2, coupled with the deter-

TABLE 1

Critical quench data for the system $BaAl_2Si_2O_8$-SiO_2

Weight percent $BaAl_2Si_2O_8$	Weight percent SiO_2	Temperature, °C	Time, hours	Phases present
		Primary phase: cristobalite		
30.0	70.0	1616	8-1/2	Glass
		1610	6	Cristobalite + glass
40.0	60.0	1518	11	Glass
		1512	5	Cristobalite + glass
45.0	55.0	1451	14	Rare cristobalite + glass
		1445	8-1/2	Cristobalite + glass
50.0	50.0	1359	48	Glass
		1350	81	Cristobalite + glass
		Primary phase: celsian or hexacelsian		
52.5	47.5	1375	23	Glass
		1359	48	Celsian + glass
55.0	45.0	1382	11	Glass
		1380	5	Rare celsian + glass
60.0	40.0	1446	11	Glass
		1434	5	Rare celsian + glass
70.0	30.0	1531	4	Glass
		1525	8-1/2	Celsian + glass
80.0	20.0	1636	7-1/2	Glass
		1632	7	Hexacelsian + glass
85.0	15.0	1600	10	Hexacelsian + celsian + glass
		1500	21	Celsian + glass
		Eutectic		
40.0	60.0	1311	50	Cristobalite + glass
		1307	60	Cristobalite + celsian
47.5	52.5	1311	50	Cristobalite + celsian
		1307	60	Cristobalite + celsian
50.0	50.0	1304	3-1/4	Cristobalite + celsian
60.0	40.0	1311	33	Celsian + glass
		1304	3-1/4	Cristobalite + celsian
70.0	30.0	1315	48	Celsian + glass
		1307	60	Cristobalite + celsian
80.0	20.0	1311	50	Cristobalite + hexacelsian + celsian

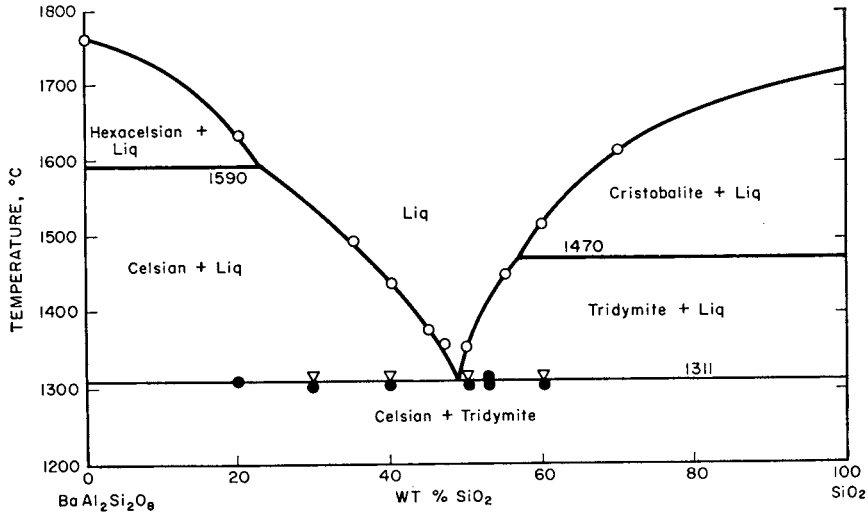

Fig. 5. Devised phase diagram for the system $BaAl_2Si_2O_8$–SiO_2.
Points on liquidus curve: o
Points above the eutectic: ▽
Points below the eutectic: ●

mination of the lowest temperature at which one of the two crystalline phases disappeared from the completely devitrified glasses. The data of table 1 show essential agreement in the results from six different compositions, in indicating that the eutectic temperature is above 1307° but below 1315°C. Strictly speaking, the determined eutectic is that between cristobalite and celsian, rather than that between tridymite and celsian. However, on the basis of many previous studies of binary systems involving SiO_2, it is believed that the metastable cristobalite-celsian eutectic would differ but little in composition or in temperature from the stable tridymite-celsian eutectic.

The coexistence of $BaAl_2Si_2O_8$ and SiO_2, as indicated in the above described relations, could not be verified at temperatures lower than 1200°C by the use of $BaAl_2Si_2O_8$–SiO_2 compositions alone. This is because glasses in this system are extremely difficult to devitrify substantially in the sub-1200° temperature range. However, unpublished results of the authors on the $BaSi_2O_5$–$BaAl_2Si_2O_8$–SiO_2 system furnished further confirmation of the coexistence of $BaAl_2Si_8O_2$ and silica at temperatures below 1200°C. Numerous tests ranging in temperature from 1000°C to 1050°C, and in time from 28 to 75 hours, invariably revealed these two compounds as compatible phases. Thus, the mullite–celsian–sanbornite and mullite–sanbornite–tridymite assemblages of figure 1 are conclusively refuted for temperatures well into the sub-eutectic region. It is believed that, short of extensive hydrothermal studies, attempts to extend the investigation to lower temperatures would be impracticable.

DISCUSSION

The present investigation provided no support for the complex relations between celsian and silica shown in figure 1, as deduced from the results of Thomas (1950) and of Toropov, Galakhov, and Bondar (1954). According to their interpretations, all compositions in the range 100 percent to 90 percent celsian (by wt) should yield celsian, sanbornite, and mullite below 1190°C (Thomas) or 1240°C (Toropov, Galakhov, and Bondar). For the composition range 90 percent celsian to 100 percent silica, all compositions should yield sanbornite, mullite, and silica below 1175°C (Thomas) or 1230°C (Toropov, Galakhov, and Bondar). Between approximately 80 percent and 50 percent celsian, a liquidus curve for mullite is indicated for both studies. In sharp contrast to these complex relations, the present study yielded simple mixtures of celsian and silica below 1311°C for the entire composition range. The liquidus relations, too, are those of a simple binary eutectic system between $BaAl_2Si_2O_8$ and SiO_2, complicated only by polymorphic inversion in each of the two end-members.

Certain aspects of the investigation by Thomas (1950) may account in part for the discrepancies between his study and the present one. As Seki and Kennedy (1964) have noted, Thomas appears to have identified celsian as mullite and hexacelsian as celsian. Furthermore, Thomas departed widely from the generally accepted practice of closely bracketing the liquidus temperatures. His limiting runs were seldom less than 40° apart, and in some instances the spread was well over 100°. The brief abstract of Wisely and Thomas (1953) suggests that the primary field of celsian in the BaO–Al_2O_3–SiO_2 system was subsequently found to be more extensive than in the original Thomas investigation. Apparently, no details of this extended field were ever published, and efforts of the present authors to obtain clarification were unsuccessful.

Toropov, Galakhov, and Bondar (1954) assigned higher values than did Thomas (by about 50°C) to several critical temperatures in the system. Compositions of certain critical points also differed by several percent in the two studies. All in all, however, there is remarkable agreement, in all essential features, between the phase relations deduced from their results and those of Thomas (fig. 1). The present authors are at a loss to explain this agreement, in view of the markedly different results of their own study. In the face of this discrepancy, extensive use was made of X-ray diffractometry, in an effort to minimize the possibility of faulty interpretation of experimental data. Every devitrified glass was subjected to careful X-ray study, and each revealed only a mixture of celsian (or hexacelsian) and cristobalite. Some interstitial glass was detected optically. Most of the non-critical runs omitted from table 1 were made with the same thought in mind, and all were subjected to X-ray analysis. Optical study, too, was used wherever practicable, but in no instance was mullite identified as a primary phase, in runs of 50 to 75 hours durations. The results in all cases were consistent with the relations shown in figure 5 and not those of figure 1.

The revised phase diagram for the system $BaAl_2Si_2O_8$–SiO_2 indicates agreement between the experimental and the natural systems, since in the laboratory, as in nature, celsian has been shown to coexist with free silica. The apparent discrepancy suggested by the findings of Thomas (1950) and of Toropov, Galakhov, and Bondar (1954) has thus been resolved. On the basis of the new diagram, too, it can be confidently predicted that the experimental study of the ternary systems $KAlSi_3O_8$–$BaAl_2Si_2O_8$–SiO_2 and $CaAl_2Si_2O_8$–$BaAl_2$–Si_2O_8–SiO_2 will be considerably less complex than previously anticipated. The new results on the $BaAl_2Si_2O_8$–SiO_2 system are compatible both with the natural phase assemblage celsian–sanbornite–quartz and with the like experimental assemblage reported by Seki and Kennedy (1964). The low-temperature stability of celsian and the high-temperature stability of hexacelsian reported by Lin and Foster (1968) and confirmed in the present study are consistent with the non-occurrence of hexacelsian in nature. The authors interpret the low-temperature hydrothermal synthesis of hexacelsian by Seki and Kennedy (1964) as due to metastability, since their own hydrothermal experiments indicated that hexacelsian changes to celsian under similar temperature conditions and more prolonged runs. A similar tendency is noted in the case of the calcium feldspar, anorthite. Here, too, metastable forms may be encountered as transitory forms, as a prelude to the crytallization of the stable anorthite.

The relations established in this study have important implications for the phase relations in the general ternary system BaO–Al_2O_3–SiO_2. The existence of a simple eutectic between celsian and tridymite, as depicted in figure 5, requires that a celsian-tridymite boundary curve should extend through this eutectic, to either side of the $BaAl_2Si_2O_8$–SiO_2 join. Such relationships have been verified by unpublished data of the authors on the system $BaSi_2O_5$–$BaAl_2Si_2O_8$–SiO_2 and by unpublished results of Semler and Foster on the system $BaAl_2Si_2O_8$–SiO_2–Al_2O_3. In these respects, also, the authors' results stand in sharp contrast to those of Thomas (1950) and of Toropov, Galakhov, and Bondar (1954), in which no $BaAl_2Si_2O_8$–SiO_2 boundary curve was indicated.

Some workers believe that feldspars take up excess silica in solid solution. The present study throws no light upon such a possibility. Should such an effect be present, the eutectic horizontal would terminate short of the celsian end-member so as to provide for an area (probably very limited in width) of celsian solid solution. Since the compositions of such solid solutions would be confined within the celsian-silica system, there would be no dip in the eutectic horizontal. This is in contrast to the gradual lowering of the eutectic horizontal shown in the system diopside ($CaMgSi_2O_6$)–anorthite ($CaAl_2Si_2O_8$), as pictured by Osborn (1942). In the latter case the solid solutions responsible for this effect are believed to extend from diopside toward calcium Tschermak's molecule ($CaAl_2SiO_6$), thus deviating in composition from the diopside-anorthite series. The possibility of limited solid solution of celsian in silica, with consequent raising or lowering of the 1470°C tridymite-

cristobalite inversion temperature, is under continuing investigation in our laboratories. Present indications are inconclusive and suggest that, because of the extreme sluggishness of reactions in the silica-rich portion of the celsian-silica system, indirect methods must be used, such as a close study of the temperature of the tridymite-cristobalite boundary curve in the neighboring $BaSi_2O_5$–$BaAl_2Si_2O_8$–SiO_2 system.

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