THE CRYSTAL STRUCTURE OF SANBORNITE, BaSi$_2$O$_5$

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ABSTRACT

Sanbornite, previously described as triclinic, is orthorhombic, space group *Pcmn*. The unit cell, $a_0 = 4.63$, $b_0 = 7.69$, $c_0 = 13.53$ Å, contains 4 BaSi$_2$O$_5$.

A structure with thirteen parameters has been found, yielding an overall residual factor R of 0.10. It is a silicate sheet structure with two (Si$_4$O$_{10}$)$^{-4}$ sheets per unit cell lying parallel to (001) at $z = 1/4$ and 3/4, and two barium ions lying roughly half way between each successive pair of sheets. Each sheet consists of continuously linked, distorted, six-membered rings of SiO$_4$ tetrahedra. Unshared vertices of tetrahedra lie on both sides of each sheet. Each barium ion is surrounded by seven near oxygen ions. Interionic distances are of expected magnitude.

INTRODUCTION

The mineral sanbornite, BaSi$_2$O$_5$, was first described by Rogers (1932). Only one occurrence is known, near Incline, Mariposa County, California,[†] where it is found with the rare mineral gillespite (BaFeSi$_4$O$_{10}$; known from only one other locality), quartz, witherite and other minerals. Sanbornite occurs as white to colorless, translucent to transparent, anhedral plates up to 2 or 3 cm. across and up to 5 mm. in thickness. Cleavage parallel to (001) is very prominent, and the cleavage lamellae are brittle and exhibit a pearly luster. Cleavage parallel to (100) is very much less prominent and cleavage parallel to (010) is indistinct.

Many fragments of sanbornite when examined between crossed polarizers show an extinction effect which Rogers interpreted as polysynthetic twinning on (010) [(100) of the present paper] with small, variable extinction angles to the trace of this plane. On this basis, lacking morphological or x-ray evidence, Rogers assigned sanbornite to the triclinic system. Rogers noted, however, that "the twinning is not as sharp and well defined as the usual albite twinning of plagioclase. The plates are usually somewhat bent and distorted, so much so that the angle between (001) and twinned (001) could not be determined with the reflection goniometer." The crystals selected for the present single-crystal x-ray examination showed no optical or x-ray evidence of twinning. The x-ray diffraction symmetry of sanbornite is orthorhombic, indicated by intensity relationships as well as metrically.

Platy crystals of BaSi$_2$O$_5$ obtained from melts were first described by

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† Three small veins, from a few inches to a few feet in width, in metasediment, about 1500 feet N. 15° W. of Trumbull Peak, NE. 1/4 Sec. 9, T. 3 S., R. 19 E., Mount Diablo base and meridian, close to 4400 feet in elevation.

Bowen (1918) as orthorhombic. Eskola (1922) also obtained $BaSi_2O_5$ crystals from melts and again believed them to be orthorhombic. To reconcile his findings with those of Bowen and Eskola, Rogers postulated dimorphism of $BaSi_2O_5$, but this has not been substantiated. In the present study sanbornite, sanbornite heated to just below the melting point*, and a sintered mixture of the composition $BaSi_2O_5$ or "$BaO \cdot 2SiO_2$" ($BaCO_3 + 2SiO_2 \cdot xH_2O$ heated at 1450° C. for $1\frac{1}{4}$ hours and at 1375° C. for $2\frac{3}{4}$ hours) gave the same x-ray powder diffraction pattern. This pattern is compared in Table 1 with d spacings calculated from cell dimensions obtained from single-crystal photographs, and with uninterpreted patterns for "$BaO \cdot 2SiO_2$" given by Austin (1947) and Thomas (1950), the latter reporting "only the most persistent peaks". An uninterpreted powder pattern for "$BaO \cdot 2SiO_2$" given by Levin and Ugrinic (1953) contains many lines for which there are no corresponding calculated spacings and is otherwise in serious disagreement with the above three patterns, so that comparison of this pattern with the others is not feasible. It seems clear that in the material of Levin and Ugrinic the preponderant phase was not $BaSi_2O_5$ of sanbornite structure.

Optical data for $BaSi_2O_5$ as reported by various investigators are summarized in Table 2.

Cell Dimensions and Space Group

Weissenberg and precession photographs obtained from several single crystals of sanbornite using

$$Cu \ (K\alpha_1 = 1.54050 \text{ Å.}, \ K\alpha_2 = 1.54434, \ K\beta_1 = 1.39217)$$

and

$$Mo \ (K\alpha_1 = 0.70926, \ K\alpha_2 = 0.71354, \ K\beta_1 = 0.63225)$$

radiation showed Laue symmetry *mmm* and gave cell dimensions $a_0 = 4.63 \pm 0.01$ Å, $b_0 = 7.69 \pm 0.02$ Å, $c_0 = 13.53 \pm 0.02$ Å; cell volume 482 Å³; $a:b:c = 0.602:1:1.759$. (The convention $c < a < b$ is not followed here for the sake of emphasizing the structural relation of sanbornite to other sheet silicates.) Systematic absences were found only for diffraction maxima with l odd in $0kl$ and with $(h+k)$ odd in $hk0$. Diffraction symbol $mmmPc$-n is thus indicated, embracing space groups $Pcmn$ (D_{2h}^{16}) and $Pc2_1n$ (C_{2v}^9).

In the hope of deciding between these two space groups, evidence for absence of a center of symmetry was sought by testing for a piezoelectric effect. No piezoelectric effect was detected with an adaptation of the Stokes (1947) modification of the Giebe-Scheibe apparatus. Crystal

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* The melting point of $BaSi_2O_5$ has been reported as 1420° C. (Eskola, 1922) and 1418° C. (Levin and Ugrinic, 1953).

TABLE 1. POWDER X-RAY DIFFRACTION PATTERNS FOR BaSi₂O₅

	Calculated			Measured		Austin (1947)[4]		Thomas (1950)[5]	
hkl	d, Å	I[1]		d, Å[2]	I[3]	d, Å	I	d, Å	I
002	6.77	18.9		6.79	30	6.7	9		
012	5.08	20.5		5.08	30				
101	4.38	0.1		—	—				
110	3.97	95.8		3.97	100	4.00	100 (1)	3.98	VS
020	3.84	4.1							
102	3.82	1.1	}10.5	3.82	10				
111	3.81	5.3							
112	3.422	47.0	}49.9	3.422	50	3.55	20		
004	3.382	2.9							
022	3.342	60.0		3.342	70	3.28	23	3.35	M
103	3.231	18.3		3.226	20				
014	3.096	50.6		3.092	75	3.14	60 (2)	3.12	S
113	2.979	4.6		2.980	5				
121	2.890	3.7		2.888	5				
104	2.731	22.5	}67.4	2.720	55	2.77	36	2.72	W
122	2.710	44.9							
114	2.574	13.0		2.574	15	2.60	10		
024	2.540	3.3		2.541	5				
123	2.473	0.0		—	—				
032	2.397	5.2		2.394	5				
105	2.336	2.7		2.327	5				
200	2.315	13.0		2.317	15	2.35	22		
201	2.282	1.4		2.281	<5				
006	2.255	0.0		—	—				
130	2.243	8.7	} 8.8	2.236	10				
115	2.235	0.1							
124	2.227	31.0		2.226	30	2.27	50 (3)	2.23	W
131	2.212	0.3		—	—				
202	2.190	11.4	}15.4	2.193	15	2.23	50		
211	2.188	4.0							
016	2.164	18.7		2.162	25	2.17	50		
132	2.129	47.2		2.130	40	2.06	25	2.13	W
212	2.107	12.0		2.109	10				
203	2.060	1.6		—	—				
034	2.043	6.8		2.038	5				
106	2.027	13.1		2.025	15				
133	2.008	0.6		—	—				
125	1.997	2.9							
213	1.989	6.4	}12.8	1.990	10				
220	1.983	3.5							
221	1.962	0.0		—	—				
116	1.960	0.0		—	—				
026	1.945	0.0		—	—				
040	1.922	12.2		1.921	10				
204	1.910	1.9		—	—	}1.91	15		
222	1.903	14.6		1.903	10				
134	1.869	1.7		—	—				
214	1.854	14.1	}20.9	1.850	20	1.87	20		
042	1.849	6.8							
223	1.815	3.0		1.815	5				
126	1.793	20.7		1.793	20	1.78	12		
				1.736	5				
				1.709	5				
				1.690	15				
				(About 50 more		1.59	10		
				lines observed)		1.48	13		
						1.46	10		
						1.29	10		

[1] Relative intensities calculated for Cu Kα radiation (λ = 1.5418 Å).
[2] Philips 114.6 mm. diameter powder camera, Straumanis mounting; 0.2 mm. diameter glass capillary with walls 0.1 mm. thick, containing mixture 50 per cent by volume each of sanbornite and powdered silica glass (to minimize the otherwise serious preferred orientation in the powder sample, arising from the prominent {001} cleavage); Cu Kα radiation.
[3] Relative peak intensities above background from densitometer measurements of powder photograph on Eastman Kodak Type AA film for which density was calibrated as a function of exposure time.
[4] BaO·2SiO₂ mixture fired twice to maximum temperature of 1430° C. and crystallized at 1400° C.; duplicate patterns obtained from two different samples, "checked petrographically and found to be homogeneous," with a Philips diffractometer using "copper K alpha radiation, filtered by nickel." Numbers in parentheses in I column indicate the three strongest maxima as noted by Austin.
[5] Thomas reported "only the most persistent peaks" summarized from several patterns obtained with a Philips diffractometer using "Cu Kα (λ = 1.5393 a. u.) radiation with a nickel filter."

TABLE 2. OPTICAL DATA FOR $BaSi_2O_5$

Eskola (1922) Rogers (1932) This paper	$BaSi_2O_5$ Sanbornite Sanbornite	$\alpha \approx b$ α-a	$\beta \approx a$ β-b	$\gamma \perp$ prominent cleavage $\gamma \approx c$ ($\frown \perp$ prominent cleavage) $\gamma = c$ ($\perp$ prominent cleavage)		
		α	β	γ	$2V_\alpha$	$'\alpha \cdot \beta \cdot \gamma)^{1/3}$
Bowen (1918)	$BaSi_2O_5$	(1) 1.595 (2) 1.598	1.613 1.617	(3)	$\sim$70° (meas.)	—
Eskola (1922) (5893 Å)	$BaSi_2O_5$	1.597	1.612	1.621	$\sim$75° (meas.) 75° (calc.)	1.610
Rogers (1932)	Sanbornite	1.597	1.616	1.624	65° (calc.)	1.612
Greene and Morgan (1941)	$BaSi_2O_5$	1.598	1.619	—	—	—
This paper (5893 Å)	Sanbornite	1.598 ±0.001	1.617 ±0.002	1.625 ±0.002	67° [meas. (4)] 65° (calc.)	1.613

Mean refractive index calculated for $BaSi_2O_5$ [using density 3.77 g. cm.$^{-3}$ calculated from sanbornite
  cell dimensions and specific refractive energies from Larsen and Berman (1934)]................   1.610
Lorentz-Lorenz refraction...   25.3 cm.$^?$

  (1) Crystals in barium-rich glass.
  (2) "Pure" crystals from $BaSi_2O_5$ melt.
  (3) Bowen reported refractive indices for the two vibration directions parallel to the platy development of
    his crystals as α and γ, the optic axial plane thus lying parallel to the plates (see his Fig. 2). He did
    not report a value for β presumably because of the thinness (ca. 0.03 mm.) of his plates, although he
    did report an optic axial angle. It seems apparent, however, that Bowen's α and γ are to be correlated
    with α and β, respectively, of the above table, in which case the optic axial plane must lie normal to
    Bowen's plates rather than parallel to them, and his crystals are platy parallel to (001).
  (4) Measured with the universal stage and by the Mallard method.

fragments suspended on fine nylon filaments and dipped into liquid
nitrogen exhibited on removal from the liquid nitrogen no electrostatic
charge. The absence of positive indication of a piezoelectric effect or
combined pyro-piezoelectric effect and the lack of morphological data
preclude a choice between the two possible space groups at this stage.

DENSITY, COMPOSITION AND CELL CONTENT

A sample of sanbornite for analysis was purified as follows: The rock
was crushed to pass 60 mesh and washed with bromoform (G. = 2.88)
to remove light minerals (principally quartz). Gillespite ($BaFeSi_4O_{10}$)
and other iron-bearing minerals were largely removed by passing the
sample repeatedly through the Frantz Isodynamic Separator (20° tilt,
1° cant, 1.5 amp.). The fraction sinking in methylene iodide (G. = 3.3)
and floating in Clerici solution (G. = 3.75) was nearly pure sanbornite.
This fraction was passed through a narrow sorting chute under the
binocular microscope and doubtful grains (some partly witherite) were
removed by hand picking with the aid of a hollow-needle, suction-
operated grain collector. The very fine material was removed in the
heavy-liquid separations. Powder patterns prepared from random sam-
ples of the material thus purified showed no extraneous lines.

The density determined pycnometrically, using toluene, on 7 g. of the
purified material is 3.71_2 g. cm.$^{-3}$. The mean density of four small (4 to

TABLE 3. ANALYSES OF SANBORNITE

	I	II	III	IV	V	VI
	Weight per cent					
SiO_2	44.13	44.19	44.16	43.92	42.2	Large amount
Al_2O_3	0.41	0.40	0.41		1.5	Small amount ($X\%$)
Fe_2O_3	0.24	0.22	0.23		tr	Very small amount ($0.X\%$)
CaO	0.20	0.20	0.20		0.1	Small amount ($X\%$)
SrO	0.08	0.08	0.08		0.2	Very small amount ($0.X\%$)
BaO	54.05	54.02	54.04	56.08	50.4	Large amount
MgO	0.27	0.29	0.28			Very small amount ($0.X\%$)
H_2O-	0.15	0.15	0.15			[Traces ($0.0X\%$ to $0.000X\%$)
H_2O+	0.52	0.56	0.54			of Na, Cr, Mn and Ti]
Total	100.05	100.11	100.09	100.00	94.4	
Density $(g. cm.^{-3})$			3.71₂	3.77	4.19	

I, II. Quantitative chemical analyses on 10 g. of purified sanbornite by Dr. R. Klemen, Vienna. Iron is reported as Fe_2O_3, no determination having been made of Fe^{+2} because of the small amount of iron present.

III. Mean of analyses I and II. Density determined pycnometrically using 7 g. of material.

IV. Calculated for $BaSi_2O_5$. Density calculated using sanbornite cell dimensions. Eskola (1922) reported a measured density for synthetic $BaSi_2O_5$ of 3.73 g. cm.⁻³.

V. Rogers (1932): "analysis made upon one gram of carefully selected material which was, however, not quite pure." "The specific gravity determined on 0.3 g. is about 4.19. The value is approximate as the mineral contained a very small amount of included minerals."

VI. Spectrographic examination of purified sanbornite by Kennard and Drake, Los Angeles (Lab #19418). Rogers (1932) reported a spectrographic examination showing Ba, Sr, Ca, Si and Al.

20 mg.) fragments determined with the Berman balance is 3.70 ± 0.02. Quantitative chemical and qualitative spectrographic analyses are presented in Table 3.

The cell dimensions and pycnometric density given above and the composition in column III of Table 3 give the unit cell content (weight of unit atomic weight 1.6602×10^{-24} g.)

$$Ba_{3.80}Ca_{0.04}Sr_{0.01}Mg_{0.07}Fe^{+3}_{0.03}Al_{0.09}Si_{7.92}O_{19.93}\cdot 0.41H_2O.$$
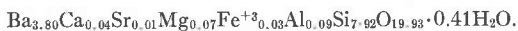
The hydrogen, reported as water, is presumably non-essential. Calcium, strontium and barium ions commonly substitute for one another in other compounds at least to a slight extent, and hence may reasonably be grouped together. The presence of the much smaller and thus seemingly extraneous cations Mg^{+2}, Fe^{+3} (perhaps present as the larger Fe^{+2}) and Al^{+3} is not explained, although aluminum ions might replace a small

proportion of silicon ions in the SiO_4 tetrahedra. This possibility will be considered more fully in the sequel on solid solution. Considering as essential only the barium, calcium, strontium, silicon and oxygen, and allowing for a small amount of quartz impurity, recalculating to twenty oxygen ions per unit cell gives

$$(Ba_{3.96}Ca_{0.04}Sr_{0.01})Si_{8.00}O_{20}$$
$$Sum = 4.00$$
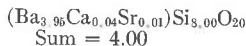
(calculated density 3.75 g. cm.$^{-3}$), the total excess matter amounting to 2.93 per cent by weight. Thus sanbornite closely approaches in composition

$$Ba_4Si_8O_{20} = 4\ BaSi_2O_5$$

(formula weight 273.48, calculated density 3.77 g. cm.$^{-3}$).

DETERMINATION OF THE STRUCTURE

Preliminary

Intensities of diffraction maxima for the three principal zones were obtained from zero-layer Weissenberg photographs made with molybdenum radiation using multiple-film technique. Intensities were estimated by visual comparison of the diffraction spots with an arbitrary scale consisting of a film of the same type developed in the same manner, bearing a series of spots varying in intensity as a function of exposure time. Lorentz and polarization factors for equatorial photographs (Buerger, 1941) were applied, and absorption corrections were applied assuming the crystals, which were roughly equant, to be spheres (Evans and Ekstein, 1952). The Bond sphere grinder could not be used because of the extremely easy (001) cleavage. Absorption corrections were small owing to small crystal size ($\sim$0.2 mm. in greatest dimension), the moderately small absorption coefficient for molybdenum radiation, and the limited angular range (θ less than 45°) of spots obtainable using molybdenum radiation. No allowance was made for extinction.

At a late stage in the structure determination the scale factor, for conversion of observed structure amplitudes H (based on the arbitrary intensity scale) to absolute values $|F_o|$, was obtained for each zone, and an overall temperature coefficient B was evaluated. These were derived through least-squares analysis of $\log_e (H/|F_{c_0}|)$ as a linear function of $\sin^2 \theta$, where F_{c_0} is the calculated structure amplitude using scattering factors for ions at rest f_0. The value of $(H/|F_{c_0}|)$ at $\sin^2 \theta = 0$ was taken as the scale factor, and the slope of the line was taken to equal $-B/\lambda^2$. The value of B thus derived (0.48 Å^2) is an overall temperature coefficient, for which it is assumed that the mean displacement is the same for all ions and all directions in the crystal.

The temperature factors were applied to the calculated structure

amplitudes F_{c_0} to give the values F_c listed with corresponding values of $|F_o|$ in Tables 4A-C.

The scattering factors f_0 used for Ba^{+2} are from values for Ba^0 adjusted by analogy with values for Ca^{+2} relative to those for Ca^0; values for Ba^0, Si^{+4} and O^{-2} are from *Internationale Tabellen* (1935). In view of the close approach of sanbornite to the ideal composition $BaSi_2O_5$, no allowances in scattering factors were made for substitution.

For lack of evidence to the contrary, it was assumed that the structure is centrosymmetric, hence the space group is *Pcmn*, and that it is not a defect structure, *i.e.*, all equivalent positions of any set are fully occupied. Thus it is required to distribute four barium, eight silicon and twenty oxygen ions among the positions (*International Tables*, 1952)

$$8 \ d \quad x, y, z; \text{ etc.}$$
$$4 \ c \quad x, 1/4, z; \text{ etc.}$$
$$4 \ b \quad 1/2, 0, 0; \text{ etc.}$$
$$4 \ a \quad 0, 0, 0; \text{ etc.}$$

TABLE 4A. OBSERVED AND CALCULATED STRUCTURE AMPLITUDES FOR $0kl$ REFLECTIONS

$0kl$	$|F_o|$	F_c	$0kl$	$|F_o|$	F_c	$0kl$	$|F_o|$	F_c
002	45*	+ 82	0, 2, 18	0	− 37	062	77	− 72
004	63	+ 63	032	89	+ 83	064	56	− 54
006	0	− 3	034	116	+110	066	0	− 7
008	183	−165	036	154	+159	058	74	+ 76
0, 0, 10	87	− 86	038	143	+134	0, 6, 10	93	+ 94
0, 0, 12	82	− 82	0, 3, 10	0	− 6	0, 6, 12	84	+ 90
0, 0, 14	91	− 83	0, 3, 12	26	+ 26	0, 6, 14	53	+ 56
0, 0, 16	0	− 10	0, 3, 14	95	− 98	072	50	+ 62
0, 0, 18	0	+ 21	0, 3, 16	87	−102	074	51	+ 58
0, 0, 20	0	+ 42	0, 3, 18	0	− 44	076	113	+119
0, 0, 22	89	+ 89	040	241	+223	078	94	+ 94
012	74*	− 80	042	123	+121	0, 7, 10	0	+ 8
014	221	−203	044	46	+ 40	0, 7, 12	0	+ 12
016	188	−174	046	0	− 10	0, 7, 14	79	− 78
018	78	− 78	048	108	− 99	080	64	+ 61
0, 1, 10	62	− 59	0, 4, 10	99	− 99	082	109	+117
0, 1, 12	35	+ 38	0, 4, 12	76	− 80	084	33	+ 29
0, 1, 14	64	+ 68	0, 4, 14	69	− 64	086	0	− 25
0, 1, 16	84	+ 83	0, 4, 16	0	− 17	088	30	− 29
0, 1, 18	77	+ 77	0, 4, 18	0	+ 18	0, 8, 10	105	−100
0, 1, 20	35	+ 36	052	41	− 44	0, 8, 12	47	− 74
020	70	− 67	054	204	−185	092	35	− 43
022	213	−205	056	108	−114	094	101	−106
024	75	− 63	058	41	− 39	096	88	− 79
026	0	+ 8	0, 5, 10	74	− 75	098	0	− 50
028	29	+ 29	0, 5, 12	56	+ 63	0, 10, 0	151	−128
0, 2, 10	180	+163	0, 5, 14	43	+ 41	0, 10, 2	0	− 41
0, 2, 12	100	+ 97	0, 5, 16	39	+ 62	0, 10, 4	0	− 31
0, 2, 14	46	+ 47	0, 5, 18	83	+ 87	0, 10, 6	0	− 10
0, 2, 16	24	+ 32	060	152	−154	0, 10, 8	68	+ 71

* Observed only on photographs made using copper radiation, for which the absorption coefficient in sanbornite is high.

TABLE 4B. OBSERVED AND CALCULATED STRUCTURE AMPLITUDES FOR $h0l$ REFLECTIONS

$h0l$	$|F_o|$	F_c	$h0l$	$|F_o|$	F_c	$h0l$	$|F_o|$	F_c
002	48*	+ 82	2, 0, 18	0	− 19	4, 0, 23	0	− 12
004	65	+ 63	2, 0, 19	0	− 30	4, 0, 24	33	+ 39
006	0	− 3	2, 0, 20	49	− 38	501	87	− 95
008	142	−165	2, 0, 21	0	− 17	502	34	− 28
0, 0, 10	80	− 85	2, 0, 22	68	− 64	503	46	− 47
0, 0, 12	89	− 82	2, 0, 23	0	+ 19	504	0	− 3
0, 0, 14	98	− 82	2, 0, 24	53	− 48	505	14	− 19
0, 0, 16	0	− 10	301	72	+ 76	506	62	− 46
0, 0, 18	0	+ 21	302	74	+ 61	507	36	+ 47
0, 0, 20	42	+ 38	303	109	+105	508	60	− 49
0, 0, 22	82	+ 89	304	48	+ 45	509	67	+ 66
0, 0, 24	53	+ 38	305	52	− 51	5, 0, 10	0	+ 11
101	0	− 6	306	102	+100	5, 0, 11	58	+ 55
102	0	− 24	307	0	− 2	5, 0, 12	29	− 25
103	97	−117	308	100	+ 96	5, 0, 13	67	+ 62
104	105	−152	309	50	− 54	5, 0, 14	54	+ 41
105	72	+ 61	3, 0, 10	0	+ 10	5, 0, 15	25	+ 28
106	123	−154	3, 0, 11	78	− 85	5, 0, 16	44	+ 38
107	0	+ 6	3, 0, 12	0	+ 2	5, 0, 17	0	− 9
108	97	− 96	3, 0, 13	29	− 26	600	0	+ 3
109	0	+ 9	3, 0, 14	72	− 65	601	0	+ 5
1, 0, 10	109	−107	3, 0, 15	22	− 21	602	44	− 33
1, 0, 11	51	+ 43	3, 0, 16	81	− 72	603	57	+ 54
1, 0, 12	70	+ 70	3, 0, 17	16	− 19	604	0	− 2
1, 0, 13	0	+ 12	3, 0, 18	34	− 38	605	75	+ 87
1, 0, 14	69	+ 66	3, 0, 19	30	+ 41	606	0	+ 18
1, 0, 15	0	+ 5	3, 0, 20	31	− 32	607	49	+ 50
1, 0, 16	86	+ 84	3, 0, 21	26	+ 35	608	0	+ 2
1, 0, 17	0	+ 10	3, 0, 22	19	+ 8	609	63	+ 66
1, 0, 18	93	+ 92	3, 0, 23	0	+ 22	6, 0, 10	0	+ 11
1, 0, 19	0	− 20	3, 0, 24	27	+ 18	6, 0, 11	0	+ 4
1, 0, 20	0	+ 14	400	43	+ 28	6, 0, 12	0	+ 5
1, 0, 21	0	− 9	401	21	− 22	6, 0, 13	44	− 53
1, 0, 22	0	+ 5	402	118	+107	6, 0, 14	0	+ 5
1, 0, 23	0	− 11	403	68	− 59	6, 0, 15	0	− 34
1, 0, 24	0	− 24	404	0	+ 10	6, 0, 16	0	+ 3
200	130	−193	405	75	− 72	6, 0, 17	56	− 70
201	59	+ 46	406	62	− 44	701	67	+ 85
202	118	−134	407	85	− 92	702	0	− 9
203	63	+ 54	408	0	− 4	703	0	+ 16
204	66	− 62	409	44	− 39	704	0	− 5
205	0	+ 10	4, 0, 10	81	− 73	705	35	+ 28
206	58	+ 50	4, 0, 11	0	− 6	706	0	− 9
207	114	+124	4, 0, 12	50	− 42	707	36	− 41
208	89	+ 92	4, 0, 13	0	+ 26	708	0	+ 4
209	0	− 1	4, 0, 14	0	− 8	709	41	− 54
2, 0, 10	69	+ 73	4, 0, 15	66	+ 73	7, 0, 10	0	+ 5
2, 0, 11	0	− 2	4, 0, 16	38	− 29	7, 0, 11	26	− 44
2, 0, 12	79	+ 79	4, 0, 17	39	+ 47	7, 0, 12	0	− 6
2, 0, 13	0	+ 13	4, 0, 18	0	+ 4	7, 0, 13	38	− 45
2, 0, 14	56	+ 58	4, 0, 19	31	+ 38	800	0	+ 8
2, 0, 15	80'	− 82	4, 0, 20	32	+ 44	805	46	− 61
2, 0, 16	0	+ 14	4, 0, 21	0	+ 14			
2, 0, 17	0	− 9	4, 0, 22	0	+ 15			

* Observed only on photographs made using copper radiation, for which the absorption coefficient in sanbornite is high.

TABLE 4C. OBSERVED AND CALCULATED STRUCTURE AMPLITUDES FOR $hk0$ REFLECTIONS

$hk0$	$|F_o|$	F_c	$hk0$	$|F_o|$	F_c	$hk0$	$|F_v|$	F_c
020	68	− 66	2, 12, 0	40	− 44	590	0	− 18
040	214	+221	2, 14, 0	41	+ 47	5, 11, 0	0	+ 17
060	167	−152	310	75	+ 83	600	0	+ 3
080	73	+ 60	330	135	−133	620	0	+ 19
0, 10, 0	127	−128	350	44	+ 38	640	0	− 11
0, 12, 0	48	+ 46	370	89	−100	660	0	− 1
0, 14, 0	49	− 63	390	52	+ 46	680	0	− 21
110	208	−219	3, 11, 0	39	− 50	6, 10, 0	0	− 3
130	130	+115	3, 13, 0	0	+ 44	6, 12, 0	0	− 7
150	216	−176	400	38	+ 28	710	0	− 8
170	82	+ 86	420	100	−107	730	0	+ 8
190	89	− 80	440	51	+ 63	750	0	− 7
1, 11, 0	79	+ 91	460	45	− 37	770	0	+ 5
1, 13, 0	41	− 35	480	66	+ 81	790	0	− 8
200	180	−193	4, 10, 0	0	− 19	800	0	+ 8
220	81	+ 81	4, 12, 0	0	+ 45	820	0	+ 31
240	153	−130	510	0	− 23	840	0	− 6
260	100	+ 88	530	78	+ 83	860	0	+ 7
280	70	− 70	550	0	+ 10	880	0	− 22
2, 10, 0	84	+ 73	570	58	+ 64	8, 10, 0	0	− 2

In silicate structures each silicon ion is surrounded by four oxygen ions arranged at the vertices of a nearly regular tetrahedron. In sheet silicates each such SiO_4 tetrahedron shares one oxygen ion with each of three other tetrahedra, leaving one unshared oxygen ion per tetrahedron, and forming continuous sheets having an overall Si/O ratio of $\frac{2}{5}$.* The fact that the Si/O ratio in sanbornite is $\frac{2}{5}$ and that the mineral has a very prominent cleavage normal to the largest cell dimension suggests that it has a silicate sheet structure with the sheets lying parallel to (001).†

From symmetrical and spatial considerations it was possible to devise a satisfactory arrangement of four SiO_4 tetrahedra in each of two undulating $(Si_4O_{10})^{-4}$ sheets per unit cell lying parallel to (001) at $z=\frac{1}{4}$ and $\frac{3}{4}$. The tetrahedra in this arrangment possessed a certain range of freedom in their orientation, and in the final structure they were found to lie within this predicted range. This postulated configuration of the sheets left sufficient space for the barium ions only between the sheets, where they might be expected to lie anyway.

Patterson projections

Two-dimensional Patterson projections on (100) and (001), shown in

* Pabst (1943) has pointed out that there are other geometrical possibilities of linking tetrahedra so as to give an Si/O ratio of 2/5 but not forming sheets.

† Rogers (1932) recognized the likelihood of sanbornite having a sheet structure and without benefit of x-ray data described a pseudohexagonal sheet structure which he pictured as "probable".

Figs. 1 and 2, respectively, were made by the photographic method of Huggins (1944). The most intense maxima are attributed to barium-barium vectors in view of the high scattering power of barium relative to silicon and oxygen ions. In the (100) projection (Fig. 1) the two most intense maxima lie at approximately $\frac{1}{2} b$, $0.08_4 c$ and $\frac{1}{2} b$, $0.41_6 c$. Because vector components parallel to c have values other than 0 and $\frac{1}{2}$, the four barium ions cannot occupy positions of sets a or b, leaving only positions of set c. Equivalent positions of set c are related by vectors whose com-
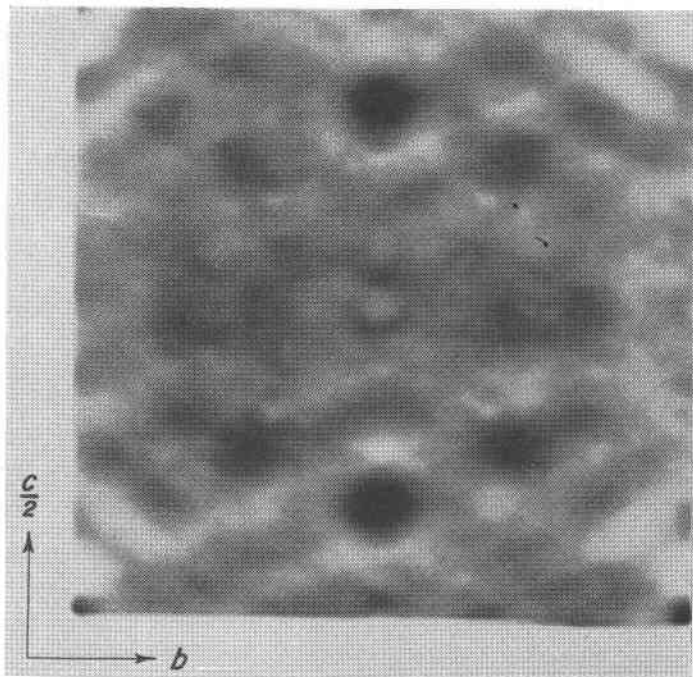
FIG. 1. Patterson projection of the sanbornite structure on (100).

ponents parallel to a, b and c may be expressed in terms of coordinates x, y and z as follows:

$$2x, 1/2, 2z; \quad 1/2, 1/2, 1/2 - 2z; \quad 1/2 - 2x, 0, 1/2.$$

The vector components parallel to c give two non-equivalent sets of four equivalent values for z_{Ba}: $(0; \frac{1}{2}) \pm \sim 0.04_2$ and $(0; \frac{1}{2}) \pm \sim 0.20_8$. In the (001) projection (Fig. 2) the one intense spot at approximately $\frac{1}{2} a$, $\frac{1}{2} b$ appears elongated parallel to a, suggesting overlap of maxima whose vector components parallel to a are close to $\frac{1}{2}$. The interequivalent-position vector components given above show that a maximum must

appear in this projection at $\frac{1}{2} a$, $\frac{1}{2} b$, but the absence of intense maxima elsewhere in the projection is explained only if $x_{Ba} = (0; \frac{1}{2}) \pm \sim\frac{1}{4}$.

Fourier projections and use of a model

Assuming the signs of $F(0kl)$ for the stronger reflections in the range k and $l = 0$ to 8 to be determined solely by the barium ions (an assumption subsequently vindicated) and taking $y_{Ba} = \frac{1}{4}$, $z_{Ba} = 0.04_2$, a Fourier projection on (100) was prepared using the strip method of Lipson and
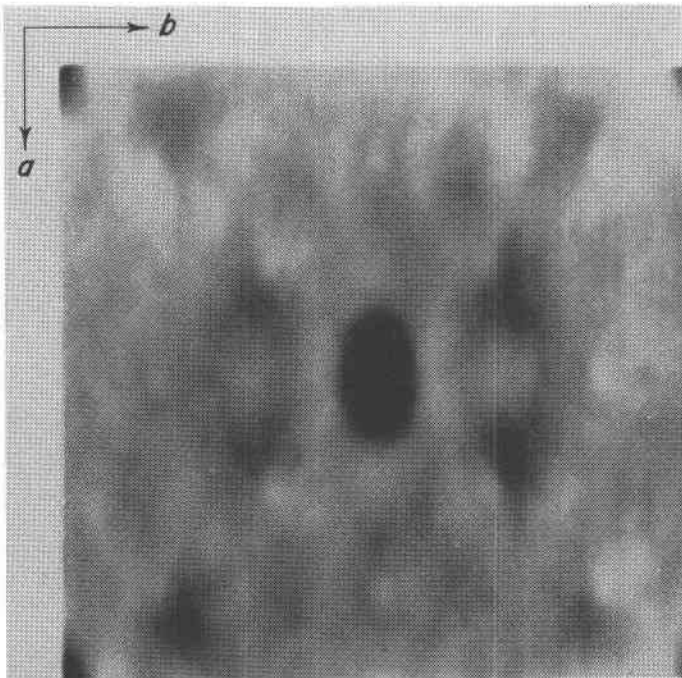
FIG. 2. Patterson projection of the sanbornite structure on (001).

Beevers (1936) with a hand calculating machine. The symmetry of the (100) projection necessitates summing only over the range 0 to $\frac{1}{4} b$ and 0 to $\frac{1}{2} c$, and any one of the four equivalent values $z_{Ba} = (0; \frac{1}{2}) \pm 0.04_2$ may be tentatively assumed. The resulting electron density map (Fig. 3) shows two large spurious peaks along $y = \frac{1}{4}$ and displacement and poor resolution of some of the expected peaks owing largely to strong termination effects. It can be shown that if z_{Ba} were taken to be one of the four equivalent values $(0; \frac{1}{2}) \pm 0.20_8$ instead of $(0; \frac{1}{2}) \pm 0.04_2$ an identical map would result but with the origin displaced $\frac{1}{4} c$; the symmetry elements of space group *Pcmn* projected on (100) are compatible with

either setting. This projection establishes, however, either that $z_{Ba} = (0; \frac{1}{2}) \pm \sim 0.04_2$ and $z_{sheets} = \frac{1}{4}$ and $\frac{3}{4}$ or that $z_{Ba} = (0; \frac{1}{2}) \pm \sim 0.20_8$ and $z_{sheets} = 0$ and $\frac{1}{2}$.

A three-dimensional working model was constructed of cardboard, assuming the SiO_4 tetrahedra to be regular tetrahedra 2.70 Å on an edge and based on the ionic distribution
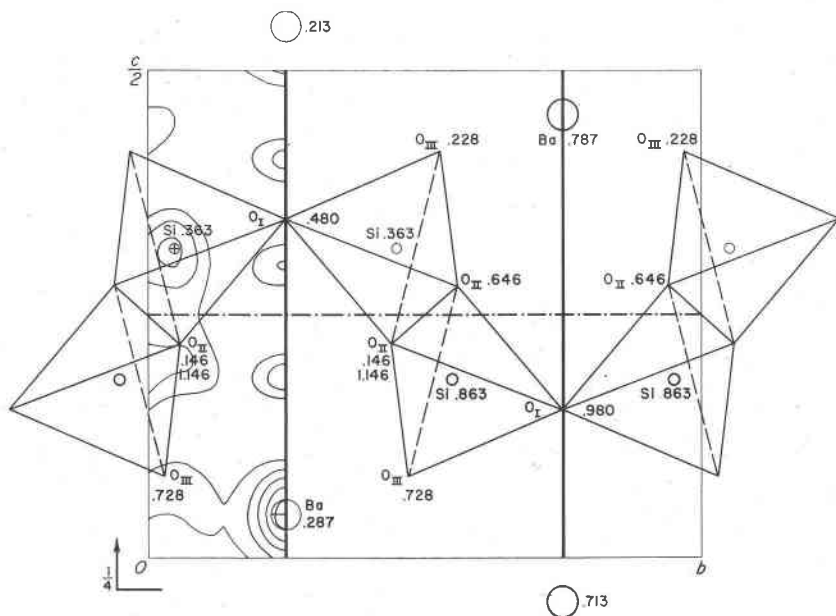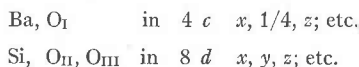
$$Ba, O_I \qquad \text{in} \quad 4 \ c \quad x, 1/4, z; \text{ etc.}$$

$$Si, \ O_{II}, O_{III} \quad \text{in} \quad 8 \ d \quad x, y, z; \text{ etc.}$$

FIG. 3. Projection of the sanbornite structure on (100). Contour interval (on an arbitrary scale) around barium ion is four times that of rest of projection.

This model aided greatly in correlating shifts in oxygen and silicon coordinates along all three axes. Using information from the (100) Patterson and Fourier projections together with trial-and-error adjustment controlled by this model, a set of approximate y and z parameters was chosen, yielding a residual factor $R(0kl) = 0.30$, where $R = \Sigma || F_o| - |F_c|| / \Sigma |F_o|$ omitting $||F_o| - |F_c||$ if $F_o = 0$. Thus it was established that $z_{sheets} = \frac{1}{4}$ and $\frac{3}{4}$, and for $y_{Ba} = \frac{1}{4}$, $z_{Ba} = (0; \frac{1}{2}) + \sim 0.04_2$.

Symmetry permits the two parallel silicate sheets per unit cell to "slide" over one another in a direction parallel to a, subject only to the condition that a shift of one sheet be accompanied by an equal but oppo-

site shift of the other. Having available approximate y and z parameters, further use of the model gave approximate tetrahedral x' parameters relative to one sheet, giving the configuration of the sheet but not its position along a. Trial-and-error shifting of x_{Ba} and of the sheets along a led to a set of parameters yielding $R(h0l) = 0.28$.

A Fourier projection on (010) was then prepared using signs of $F(h0l)$ given by the x and z parameters determined so far. It is only necessary to sum over the range 0 to $\frac{1}{2}$ along both axes. From the parameters given by this projection were obtained the signs of terms previously of uncertain or incorrect sign, and these terms were included in a new projection (Fig. 4). From this refined projection were taken, after further trial-and-error adjustment, the final x and z parameters.

From the x, y and z parameters thus derived (Table 5) have been calculated the structure amplitudes F_c (to which have been applied temperature factors) listed in Tables 4A–C with corresponding values of $|F_o|$. These calculated and observed structure amplitudes yield the residual factors (omitting terms for which $F_o = 0$) $R(0kl) = 0.069$, $(Rh0l) = 0.135$, $R(hk0) = 0.098$, and an overall value (221 terms) of 0.101. These acceptably small residual factors and the generally satisfactory agreement between calculated and observed structure amplitudes suggest that the coordinates are known with sufficient accuracy to firmly establish the structure.

DISCUSSION OF THE STRUCTURE

The structure of sanbornite given by the coordinates listed in Table 5 is shown projected on (100), (010) and (001) in Figs. 3, 4 and 5, respectively. In these figures the larger circles represent barium, the smaller circles silicon and the vertices of the outlined tetrahedra oxygen ions. The unit cell or indicated fraction thereof is outlined, and minimum symmetry elements are shown.

The oxygen ions of type I lie in the m planes, which are parallel to (010) at $y = \frac{1}{4}$ and $\frac{3}{4}$. Each of these oxygen ions is shared by two SiO_4 tetrahedra which are thus mirror images of one another. Each tetrahedron of such a mirror-image pair shares its two oxygen ions of type II with two other tetrahedra, each of which in turn belongs to another mirror-image pair (best seen in Fig. 5). The one oxygen ion of type III per tetrahedron is unshared, and as to be expected, the shortest silicon-oxygen distance in the tetrahedra (Table 6) is that between the silicon and this unshared oxygen ion. Adjacent mirror-image pairs are related by diagonal glide reflection across one of the two n planes parallel to (001) at $z = \frac{1}{4}$ and $\frac{3}{4}$. This results in continuously linked, distorted, six-membered rings of tetrahedra, forming undulating sheets of indefinite extent which lie parallel to (001) at $z = \frac{1}{4}$ or $\frac{3}{4}$ and have an overall Si/O

TABLE 5. FINAL COORDINATES OF IONS IN SANBORNITE

(Expressed as fractions of cell edges)

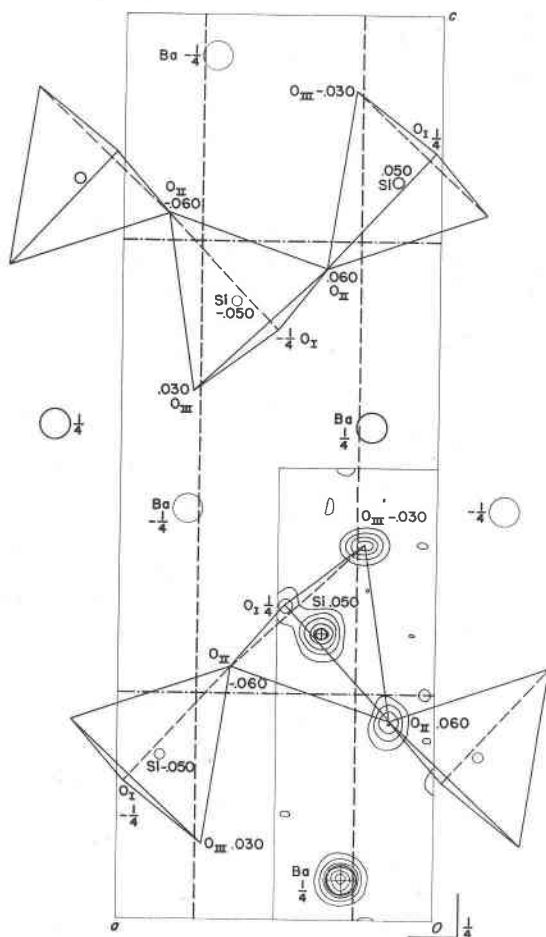|		x	y	z
Ba	in 4 c	0.287	1/4	0.045
Si	in 8 d	0.363	0.050	0.317
O_I	in 4 c	0.480	1/4	0.348
O_{II}	in 8 d	0.146	0.060	0.220
O_{III}	in 8 d	0.228	0.970	0.416

FIG. 4. Projection of the sanbornite structure on (010). Contour interval (on an arbitrary scale) around barium ion is four times, around silicon ion two times, that of rest of projection.

FIG. 5. Projection of the sanbornite structure on (001).

TABLE 6. INTERIONIC DISTANCES IN SANBORNITE

Edges of SiO₄ tetrahedra	
$O_I - O_{II}$	2.67 Å
$O_I - O_{II}$	2.74
$O_I - O_{III}$	2.62
$O_{II} - O_{II}$	2.62
$O_{II} - O_{III}$	2.68
$O_{II} - O_{III}$	2.77
Shortest intralayer intertetrahedral O–O distance:	
$O_{II} - O_{II}$	2.92
Shortest interlayer O–O distance:	
$O_{III} - O_{III}$	3.14
$Si - O_I$	1.68
$Si - O_{II}$	1.64
$Si - O_{II}$	1.65
$Si - O_{III}$	1.60
$Ba - O_I$	2.94
$Ba - O_{II}$	2 at 2.86
$Ba - O_{III}$	2 at 2.74
$Ba - O_{III}$	2 at 2.77
$Ba - O_{III}$	2 at 3.14
Next shortest Ba–O distance	
$Ba - O_I$	4.20

ratio of $\frac{2}{5}$. Glide reflection across the c planes, which are parallel to (100) at $x=\frac{1}{4}$ and $\frac{3}{4}$, produces two $(Si_4O_{10})^{-4}$ sheets per unit cell lying parallel to (001) at $z=\frac{1}{4}$ and $\frac{3}{4}$. Unshared vertices of tetrahedra (oxygen ions of type III) point out in pairs alternately from one side and then the other of each sheet.

The four barium ions per unit cell lie in equal numbers roughly half way between each successive pair of sheets. Each barium ion is surrounded by seven near oxygen ions (Table 6) of which the four nearest, as to be expected, are unshared oxygen ions of type III, two belonging to

TABLE 7. COMPARISON OF SOME INTERIONIC DISTANCES

Structure	Edges of SiO$_4$ tetrahedra	Si–O	Ba–O
Sanbornite, BaSi$_2$O$_5$ (This paper)	6, 2.62–2.77 Å	4, 1.60–1.68 Å	7, 2.74–2.94 Å 2, 3.14 Å
Gillespite, BaFeSi$_4$O$_{10}$ (Pabst, 1943)	6, 2.58–2.67 Å	4, 1.56–1.64 Å	2.73, 2.98 Å
β-Na$_2$Si$_2$O$_5$ (Grund, 1954)	12, 2.50–2.75 Å	8, 1.50–1.65 Å	
Paracelsian, BaAl$_2$Si$_2$O$_8$ (Smith, 1953)			7, 2.69–2.85 Å 2, 3.33 Å
Tetragonal BaTiO$_3$ (Evans, 1951)			2.782, 2.826, 2.896 Å
Sum of ionic radii (*Internationale Tabellen*, 1935)		1.59 Å (four coordination)	2.75 Å (six coordination)

the superjacent sheet and two to the subjacent sheet.* The barium ions satisfy the excess negative charge of the sheets and thus hold them together.

The interionic distances in sanbornite (Table 6) are in satisfactory agreement with corresponding distances found in other structures as compared in Table 7.

RELATED STRUCTURES

The mineral gillespite (BaFeSi$_4$O$_{10}$) is closely related to sanbornite (Ba$_2$Si$_4$O$_{10}$) in composition and occurrence, and also possesses a silicate

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* Smith (1953) also found seven near oxygen ions about each barium ion in paracelsian. BaAl$_2$Si$_2$O$_8$.

sheet structure (Pabst, 1943), but the two structures have important differences. Gillespite is tetragonal with two *double* sheets per unit cell made up of linked, *four*-membered rings of SiO_4 tetrahedra. The barium ions in gillespite, as in sanbornite, lie between the sheets and are associated with oxygen ions of both adjacent sheets, but each ferrous ion in gillespite is associated with oxygen ions of only one sheet.

Schaller (1929) and Pabst (1943, 1955) found that the iron and barium can be completely leached out of gillespite with hydrochloric acid without complete physical disintegration. The initially bright-red flakes become colorless and the density and mean refractive index drop markedly, but the optical character and outward shape are retained, and Laue and precession photographs show that the essential silicate sheet structure persists. Sanbornite treated with even dilute hydrochloric acid quickly swells up into low-birefringent, x-ray-amorphous shreds. The greater stability of the gillespite sheet structure on leaching may arise from its having two cations, beside silicon, which are differently linked to the silicate sheets; these might be selectively removed in successive stages of leaching. Possibly the double sheets of four-membered rings of tetrahedra in gillespite are mechanically more rigid than the single sheets of six-membered rings of tetrahedra in sanbornite.

The only silicates having the composition $R_2Si_2O_5$ known to the writer are those in which $R = Ba_{1/2}$, Li, Na, K, Rb or Cs (Ramberg, 1954). It is particularly interesting that $CaSi_2O_5$ and $SrSi_2O_5$, which might be expected to be isomorphous with sanbornite, are not known. Comparison of simple disilicates for which sufficient x-ray data are available (Table 8) reveals some striking similarities. However, the crystal structures of only two of these, sanbornite and β-$Na_2Si_2O_5$, have been determined. The β-$Na_2Si_2O_5$ structure (Grund, 1954), although monoclinic, resembles the sanbornite structure in that it consists of two undulating, single $(Si_4O_{10})^{-4}$ sheets per unit cell lying parallel to the very prominent cleavage [in this case (100)], with the larger cations lying in equal numbers between each successive pair of sheets and associated with oxygen ions of both sheets. The arrangement of SiO_4 tetrahedra in the sheets is quite different, although unshared tetrahedral vertices point out alternately from both sides of each sheet as in sanbornite. Four of the eight sodium ions per unit cell are surrounded by six near oxygen ions, the remainder by five.

In summary, sanbornite, gillespite, β-$Na_2Si_2O_5$ and possibly all of the other alkali disilicates have in common the following features distinguishing them as a group from all other known phyllosilicates: (1) they contain no hydrogen (unique to this group); (2) successive sheets are held together by the larger cations (unique except for micas, brittle

micas and apophyllite); and (3) tetrahedral vertices point out from both sides of the sheets (unique except for apophyllite). Gillespite may be regarded as comprising a subgroup because (1) the sheets are double and (2) it contains more than one cation beside silicon, one of which is associated with only one sheet.

Solid Solution

As mentioned in the preceding section, no calcium or strontium analog of sanbornite is known. The analyses of sanbornite (Table 3) show very small amounts of calcium and strontium which are presumably replacing

TABLE 8. COMPARISON OF SOME PHASES OF COMPOSITION $R_2Si_2O_5$

(Crystallographic elements oriented for comparison with β-$Na_2Si_2O_5$)

Cell content	Space group	a_0	b_0	c_0	β	Prominent cleavage	Volume per O^{-2}
$Ba_4Si_8O_{20}$ (sanbornite)	$Pnam$	13.53 Å	4.63 Å	7.69 Å	—	{100}	24.1 Å³
α-$Na_8Si_8O_{20}$	(1) $Pnam$	15.45	4.909	6.428	—	{100}	24.4
β-$Na_8Si_8O_{20}$	(1) $P2_1/a$	12.307	4.849	8.124	104.12°	{100}	23.5
	(2) $P2_1/a$	12.26	4.80	8.07	104°18′	{100}	23.0
$Li_8Si_8O_{20}$	(1) $Bbmb$ or $Bb2b$	14.66	4.806	5.80	—	{100}	20.4

(1) Donnay and Donnay (1953).
(2) Grund (1954).

barium, but the limits of such substitution have not been assessed. Determination of these limits seems worthy of further study, especially with regard to strontium.

The system $BaSi_2O_5-Na_2Si_2O_5$ was investigated by Greene and Morgan (1941) who found only a simple eutectic (32 weight per cent $BaSi_2O_5$, 797° C.) and no intermediate compounds or mix crystals. Spectrographic analyses of sanbornite show virtually complete absence of the alkali elements, but the surrounding rocks are low in these elements also. In a limited substitution of two alkali ions for one barium ion in the sanbornite structure, one alkali ion could possibly occupy each former barium position, but there are apparently no holes elsewhere in this structure which can suitably accommodate the other alkali ion.

In a study of the system $BaO-SiO_2$ Eskola (1922) reported a "complete series of solid solutions" between the "very perfectly isomorphous" end members $BaO \cdot 2SiO_2$ ($BaSi_2O_5$) and $2BaO \cdot 3SiO_2$ or $BaO \cdot 1.5SiO_2$. Thomas (1950) did not consider $BaO \cdot 1.5SiO_2$ to be a compound, and extended the range of solid solution to $BaO \cdot \sim 1.4SiO_2$. Levin and Ugrinic

(1953) concluded that $BaO \cdot 1.5SiO_2$ was a compound and stated that "$BaO \cdot 2SiO_2$ and $2BaO \cdot 3SiO_2$ show unusual behavior for refractory oxide systems in that they form a complete isomorphous solid solution series without a maximum or a minimum", but they also stated that optical properties and powder patterns of the primary phase crystals did not differ significantly from those of pure $BaO \cdot 2SiO_2$ (however, see observations regarding their powder pattern in the introductory section of the present paper). Complete solid solution between $BaO \cdot 2SiO_2$ and $2BaO \cdot 3SiO_2$ was also reported by Toropov, Galakhov and Bondar (1955). It seems certain from the physical properties given that Bowen (1918), Eskola (1922), Greene and Morgan (1941), Austin (1947), Thomas (1950), and Toropov, Galakhov and Bondar (1955) all obtained $BaSi_2O_5$ of sanbornite structure on heating a mixture of this composition, as did the writer. However, it is difficult to envisage in the sanbornite structure a reasonable mechanism of removal, substitution or addition of silica or baryta which could account for such serious apparent deficiencies in silica (up to one fourth) or excesses in baryta (up to one third) as have been reported.

Thomas (1950) reported a limited solid solution between $BaO \cdot 2SiO_2$ ($BaSi_2O_5$) and $BaO \cdot Al_2O_3 \cdot 2SiO_2$ ($BaAl_2Si_2O_8$, celsian) to the extent of $BaO \cdot \sim 0.22Al_2O_3 \cdot 2SiO_2$, and another solid solution between $BaO \cdot 1.5SiO_2$ and $BaO \cdot Al_2O_3 \cdot 2SiO_2$. Similar conclusions were drawn by Toropov, Galakhov and Bondar (1955) in a reinvestigation of this same portion of the system baryta-alumina-silica. In accounting for these findings regarding $BaSi_2O_5$, Al^{+3} ions might be expected to replace some of the Si^{+4} ions in the SiO_4 tetrahedra, as is commonly the case in aluminosilicates. The resulting deficiency in positive charge might be compensated in one or both of two ways: (1) Substitutions of Al^{+3} for Ba^{+2} equal in number to the substitutions of Al^{+3} for Si^{+4} in the tetrahedra. Such replacement would probably be very limited considering the great disparity in size between the barium ion and all the common trivalent cations. (2) Accomodation of one aluminum ion in some hole in the structure for each three substitutions of aluminum for silicon ions in the tetrahedra.

Clearly these solid solutions involving the sanbornite structure need reinvestigation with attention to structural implications.

ACKNOWLEDGMENTS

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