phenyl group. The Cl—C distances (1·744 and 1·748 Å) are in agreement with those found for Cl—C (sp^2) bonds. The packing of the molecule is shown in Fig. 4. No intermolecular distances are less than 3·4 Å.

We thank Dr Rolf Göthe at the Wallenberg Laboratory, Stockholm, for providing the crystals, Professor Peder Kierkegaard for the facilities placed at our disposal, Dr Don Koenig for correcting the English, and the referee for valuable suggestions concerning the manuscript. This investigation has been financially supported by the Swedish Natural Science Research Council.

References

Brown, I. D. (1976). *Acta Cryst.* A**32**, 24–31.
DeLacy, T. P. & Kennard, C. H. L. (1972*a*). *J. Chem. Soc. Perkin* II, pp. 2141–2147.
DeLacy, T. P. & Kennard, C. H. L. (1972*b*). *J. Chem. Soc. Perkin* II, pp. 2148–2153.
Frear, E. H. (1948). *Chemistry of Insecticides, Fungicides and Herbicides*, 2nd ed., pp. 57–88. New York: Van Nostrand.
Gunther, F. A., Blinn, R. C., Carman, G. E & Metcalf, R. L. (1954). *Arch. Biochem.* **50**, 504–505.
Heijden, S. P. N. van der, Griffith, E. A. H., Chandler, W. D. & Robertson, B. E. (1975). *Canad. J. Chem.* **53**, 2084–2092.
Holan, G. (1969). *Nature, Lond.* **221**, 1025–1029.
International Tables for X-ray Crystallography (1974). Vol. IV, pp. 72–102. Birmingham: Kynoch Press.
Metcalf, R. L. (1973). *J. Agric. Food Chem.* **21**, 511–519.
O'Brien, R. D. (1967). *Insecticides, Action and Metabolism*, pp. 108–132. New York: Academic Press.
Riemschneider, R. D. & Otto, H. D. (1954). *Z. Naturforsch.* **9b**, 95–110.
Rogers, E. F., Brown, H. D., Rasmussen, I. M. & Heal, R. E. (1953). *J. Amer. Chem. Soc.* **75**, 2991–2999.

Acta Cryst. (1977). B**33**, 381–385

The Crystal Structure of $Rb_2Be_2Si_2O_7$

By R. Alan Howie and Anthony R. West

University of Aberdeen, Department of Chemistry, Meston Walk, Aberdeen AB9 2UE, Scotland

(*Received* 1 *June* 1976; *accepted* 6 *July* 1976)

The structure of the Rb beryllosilicate, $Rb_2Be_2Si_2O_7$, has been solved from three-dimensional single-crystal X-ray diffraction data obtained with a linear diffractometer. The orthorhombic unit cell, $a = 8{\cdot}92\,(1)$, $b = 8{\cdot}32\,(1)$, $c = 5{\cdot}15\,(1)$ Å, contains two formula units of $Rb_2Be_2Si_2O_7$. The structure, refined in the space group *P*2*nn* by block-diagonal least squares ($R = 5{\cdot}4\%$), is that of an infinite beryllosilicate framework, with large cavities containing 13 and 14-coordinate Rb (Rb—O = 2·85–3·83 Å). The framework consists of Si_2O_7 units whose non-bridging O atoms form the corners of planar BeO_3 groups. Mean Be—O and Si—O bond distances are 1·55 and 1·59 Å. The occurrence of several related Rb and Cs beryllosilicate phases is reported.

Introduction

During a study of compound formation in A_2O—BeO—SiO_2 systems, a new orthosilicate phase, Rb_2BeSiO_4, was encountered. A single crystal picked from a bulk preparation of Rb_2BeSiO_4 had a geometrically hexagonal pseudocell which was similar in size to the hexagonal unit cell of tridymite. On the supposition that this phase might be a novel type of filled tridymite structure, a full crystallographic study was undertaken. During the analysis, it soon became clear that the composition of the crystal was different from that of the bulk preparation and instead, corresponded to the formula $Rb_2Be_2Si_2O_7$. This had been overlooked previously because Rb_2BeSiO_4 and $Rb_2Be_2Si_2O_7$ (a bulk preparation was subsequently made) have very similar powder patterns, both of which could be indexed on similar-sized orthorhombic, geometrically hexagonal cells. The crystal structure of $Rb_2Be_2Si_2O_7$ is reported here. It bears no relation to tridymite and instead, contains unusual, planar BeO_3 units [Howie & West (1976), preliminary note].

$Rb_2Be_2Si_2O_7$ appears to be a thermodynamically stable phase, although loss of rubidium oxide by volatilization occurs above 1000–1100 °C. Up to 850 °C, however, differential thermal analysis gives no indication of chemical or structural change.

Experimental

Crystal data

A single crystal of $Rb_2Be_2Si_2O_7$ was selected from a sinter of approximate composition Rb_2BeSiO_4 which had been heated at 1000°C for 3 d. Although geometrically hexagonal ($a = \sqrt{3}c$), the unit cell of $Rb_2Be_2Si_2O_7$ is orthorhombic, $a = 8{\cdot}92\ (1)$, $b = 8{\cdot}32\ (1)$, $c = 5{\cdot}15\ (1)$ Å (refined from indexed powder diffraction data), and contains two formula units (unit-cell volume 382·2 Å^3, F.W. 357·3, $d_c = 3{\cdot}11$, $d_o^* = 3{\cdot}2$ g cm^{-3}). The space group adopted for the final solution of the structure was *P2nn* (non-standard setting of *Pnn*2, No. 34). The *hkl* reflexions with $h + k + l$ odd are either very weak or absent, giving a strong pseudosymmetry with apparent space group (consistent with the Patterson map) *I2mm* (non-standard setting of *Imm*2, No. 44).

Intensity data

Intensity data for layers *hk*0–6 were collected from a crystal approximately 50 × 50 × 150 μm, using a Hilger & Watts Y-190 linear diffractometer with Mo $K\alpha$ radiation. The ω scan of the instrument was 2·25° and each count cycle was of 1 min duration, apportioned equally between the peak count and two background counts on either side of the peak. Using the balanced-filters facility, four count cycles, which reduce to two estimates of the nett intensity, were measured for each reflexion. No absorption correction was applied.

Only reflexions for which $h + k + l$ was even were accepted initially. The data base was then reduced by averaging the intensities of *hkl* and $h\overline{kl}$ reflexions, and structure amplitudes (F_o) obtained in the usual way for 596 planes (list L1) for which the indices were either all positive or *h* only was negative. Of these, 139 reflexions were classed as unobserved ($F \leq 3 \times$ e.s.d., where the standard deviation was based on counting statistics) and were excluded from the refinement. A second structure amplitude list (L2) was obtained by averaging data for *hkl* and $\bar{h}kl$ in the first list to yield 319 reflexions (all indices positive) of which 78 were classed as unobserved and rejected.

Programs

The crystallographic programs were those of Ahmed and Barnes of the National Research Council of Canada, adapted for use on an ICL 4-70 computer by J. S. Knowles of the Computing Centre of the University of Aberdeen. The least-squares program employs the block-diagonal approximation. The weighting scheme used was initially $\sqrt{w} = |F_o|/K$ ($F_o \leq K$) or $\sqrt{w} = K/|F_o|$ ($F_o > K$) where K was set to 10·0 on the absolute scale, and finally $\sqrt{w} = 1/\{1 + [(|F_o| - K_2)/K_1]^2\}^{1/2}$, where K_1 and K_2 were set to 40·0 and 60·0. In this program the correction for anisotropic thermal vibration takes the form: $\exp[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{23}kl + B_{13}hl + B_{12}hk)]$. Standard deviations on refined parameters are estimated as: $[A_{ii}^{-1}\ \Sigma\ w\Delta F^2/(m-n)]^{1/2}$ where A_{ii}^{-1} is the diagonal element of the inverse normal equations matrix corresponding to the *i*th parameter, *m* is the number of independent reflexions (observations) and *n* is the number of parameters refined.

Atomic scattering factors, including curves for the real and imaginary components for Rb to permit correction for anomalous dispersion with data list L1, were taken from *International Tables for X-ray Crystallography* (1968).

Solution of the structure

In the initial stages, the formula of the crystal was taken to be Rb_2BeSiO_4. The Rb atoms were readily placed in the positions 2(*a*) and 2(*b*) of *I2mm*, as indicated by a Patterson map prepared from data list L2. Patterson superposition produced a choice of positions for Si and O, but with only two Si per unit cell (density measurements gave $Z = 2$), the Si positions [4(*d*) in *I2mm*] could only be half occupied. This gave a negative temperature factor for Si indicating that the sites were probably fully occupied. It seemed probable then that the assumed formula was incorrect. Consideration of the possible sites for Si, O and Be indicated $Rb_2Be_2Si_2O_7$ as the likely formula and this gave both a positive temperature factor for Si and a marked improvement in the *R* value. This model, with Be, Si in 4(*d*) and O in 8(*e*), 4(*d*) and 2(*a*), refined to a conventional *R* value of 7·9% with data L1, allowing for anomalous dispersion by Rb and with Rb, Si and O vibrating anisotropically.*

At this stage it was noted that (1) some bond lengths were unrealistic, *e.g.* Si–O 1·53 Å; (2) the O positions appeared disordered from a Fourier map – a disordered model in *I2mm* with twice the number of half-occupied O positions improved *R* to 7·2% and permitted the selection of more reasonable Si–O bond distances; (3) a few very weak reflexions

---

* Displacement of CCl_4 in a specific gravity bottle. Reproducible results were difficult to obtain, particularly with gas pycnometry, which gave apparently time-dependent densities; see discussion below.

* The validity of the correction for anomalous dispersion was tested by comparison of the *R* values for (1) parameters *xyz* with anomalous dispersion correction, (2) parameters *xyz* without anomalous dispersion correction and (3) parameters $\bar{x}yz$ with anomalous dispersion correction. The *R* value of model 1 was found to be better than those for models 2 and 3 by 0·5 and 2·0% respectively.

with $h + k + l$ odd were present in some precession and Weissenberg photographs.

Further refinement was undertaken, therefore, in the space group *P2nn*. In this space group a solution was obtained which gave satisfactory bond distances, and, excluding three reflexions for which $\Delta F/F$ was in excess of 0·6, the final R on 454 reflexions of list L1 was 6·88%. The same parameters, ignoring anomalous dispersion, give $R = 5{\cdot}4\%$ over 241 reflexions of list L2.

A few weak reflexions with $h + k + l$ odd were added to list L1 but were used only in the preparation of Fourier and difference maps. The final difference map showed no peak larger than 1·5 e Å^{-3} (4% of the Si peak and 16% of the Be peak). The Fourier map showed appropriate electron densities for Rb, Si and Be, but for the O atoms only $\frac{1}{3}$ to $\frac{1}{2}$ of the expected electron densities. The O atom peaks were much more diffuse than those of the cations.

After final refinement with L1, including anomalous dispersion, O(1) exhibited a negative value for B_{33}. To obtain the corresponding B_{iso} value for O(1) (Table 1), B_{33} was set positive by the addition of one e.s.d. However, the same atomic parameters, refined with data list L2, ignoring anomalous dispersion, gave positive values for all B_{ii}. Despite the low electron densities at the O positions, it is suggested that the significant features of the structure are well enough defined. The distribution of weighted residuals ($\Sigma\, w\Delta F^2$) in terms of $|F_o|$ and

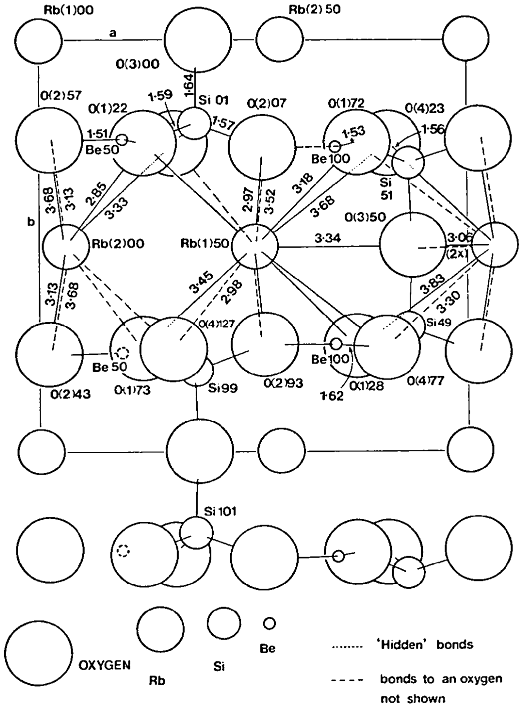

Fig. 1. (001) projection of $Rb_2Be_2Si_2O_7$.

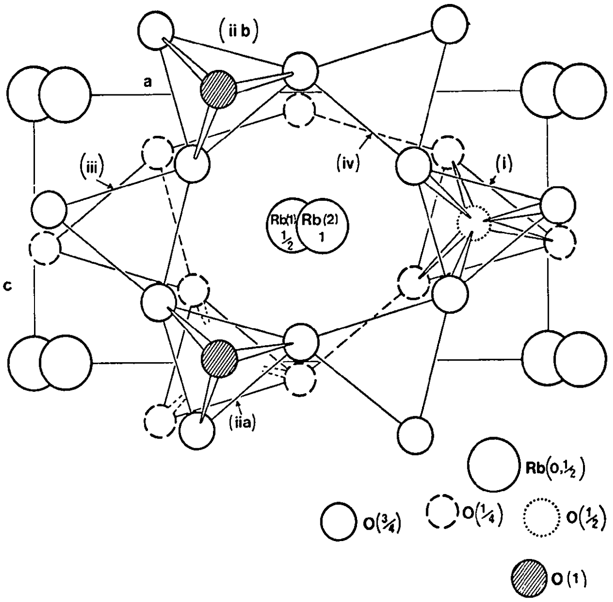

Fig. 2. Slightly idealized (010) projection of $Rb_2Be_2Si_2O_7$.

Table 1. *Atomic parameters for* $Rb_2Be_2Si_2O_7$

Estimated standard deviations are in parentheses. Positional parameters and B_{ij} are $\times 10^4$.

	N*	x	y	z	B_{iso}†	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
Rb(1)	2(*a*)	0	0	0	1·99 (5)	64 (3)	62 (4)	209 (15)	135 (45)		
Rb(2)	2(*b*)	590 (4)	5000	0	1·88 (4)	62 (3)	53 (4)	206 (14)	90 (57)		
O(3)	2(*a*)	3744 (32)	0	0	3·9 (5)	199 (49)	58 (39)	367 (192)	−220 (310)		
Be	4(*c*)	6850 (39)	7414 (37)	0 (352)	2·4 (5)						
Si	4(*c*)	3595 (6)	1965 (7)	103 (37)	1·02 (7)	29 (5)	49 (6)	74 (18)	29 (67)	−14 (60)	−14 (11)
O(1)	4(*c*)	2380 (17)	2506 (25)	2177 (35)	1·8 (3)	17 (22)	149 (30)	−10 (70)‡	51 (74)	0 (52)	−11 (31)
O(2)	4(*c*)	5204 (26)	2631 (27)	697 (34)	2·3 (4)	43 (20)	125 (26)	207 (92)	189 (86)	53 (78)	14 (33)
O(4)	4(*c*)	3050 (25)	2483 (23)	−2656 (67)	2·8 (5)	54 (26)	118 (27)	327 (110)	93 (110)	−18 (99)	−95 (41)

* Number of positions and Wyckoff notation.
† Units of B_{iso} are Å^2.
‡ A negative value for B_{ii} is meaningless – see text.

Table 2. *Representative bond distances* (Å) *and angles* (°) *for* $Rb_2Be_2Si_2O_7$

Si_2O_7		BeO_3	
Si–O(1)	1·59 (2)	Be–O(1)	1·53 (2)
Si–O(2)	1·57 (2)	Be–O(2)	1·51 (4)
Si–O(3)	1·64 (1)	Be–O(4)	1·62 (4)
Si–O(4)	1·56 (4)	O(1)–O(4)	2·73 (4)
O(1)–O(4)	2·56 (4)	O(2)–O(4)	2·68 (3)
O(1)–O(2)	2·63 (3)	O(1)–O(2)	2·66 (3)
O(1)–O(3)	2·66 (2)	O(1)–Be–O(4)	120 (2)
O(2)–O(3)	2·57 (3)	O(1)–Be–O(2)	122 (2)
O(2)–O(4)	2·59 (3)	O(2)–Be–O(4)	118 (2)
O(3)–O(4)	2·55 (3)		
O(1)–Si–O(4)	109 (1)		
O(1)–Si–O(2)	113 (1)		
O(1)–Si–O(3)	111 (1)		
O(2)–Si–O(3)	107 (1)		
O(2)–Si–O(4)	111 (1)		
O(3)–Si–O(4)	106 (1)		
Si–O(3)–Si	171 (1)		
$Rb(1)O_{13}$		$Rb(2)O_{14}$	
Rb(1)–O(1)	3·18 (2)	Rb(2)–O(1)	2·85 (2)
Rb(1)–O(1′)	3·45 (2)	Rb(2)–O(2)	3·13 (2)
Rb(1)–O(2)	2·97 (2)	Rb(2)–O(3)	3·06 (2)
Rb(1)–O(3)	3·34 (3)	Rb(2)–O(1′)	3·83 (2)
Rb(1)–O(4)	2·98 (3)	Rb(2)–O(2′)	3·68 (2)
Rb(1)–O(4′)	3·68 (2)	Rb(2)–O(4)	3·30 (2)
Rb(1)–O(2′)	3·52 (2)	Rb(2)–O(4′)	3·33 (2)

$\sin^2\theta$ indicates that the final weighting scheme is satisfactory.* An (001) projection of the structure is shown in Fig. 1 and a more schematic, slightly idealized (010) projection in Fig. 2. Bond lengths and angles are given in Table 2.

Description of the structure

The essential features of the structure are indicated in Fig. 2. The most obvious structural components are Si_2O_7 groups (i and ii). The bridging O atom of each of these groups lies on a twofold axis, either at $y=\frac{1}{2}$ (i) or at $y=0, 1$ (ii). Each terminal O atom of a Si_2O_7 group is shared with a different BeO_3 group (*e.g.* iii) to form an infinite beryllosilicate framework of empirical formula $[Be_2Si_2O_7]^{2-}$. These shared O atoms, at heights $y=\frac{1}{4}$ and $\frac{3}{4}$, approximately, form irregular six-membered rings (iv) and the Rb atoms lie in cages created by two adjacent six-membered O rings. Rb(1) at $y=\frac{1}{2}$ has, as an additional nearest-neighbour O atom, the bridging O of Si_2O_7 (i), also at $y=\frac{1}{2}$, , to complete its 13-fold coordination. Rb(2) at $y=1$ (say) has, instead, two additional nearest-neighbour O atoms which are the bridging O atoms of Si_2O_7 groups (ii*a*) and (ii*b*), and is 14-coordinate.

Details of the structure and discussion

Triangular coordination for Be is uncommon and only three other reported occurrences of BeO_3 groups are known to the authors (Y_2BeO_4, Harris & Yakel, 1967; $Ca_{12}Be_{17}O_{29}$, Harris & Yakel, 1966; K_2BeO_2, Kastner & Hoppe, 1974). Beryllosilicates are normally tetrahedral framework structures in which Be has a similar role to Al in aluminosilicates. The O–Be–O angles in $Rb_2Be_2Si_2O_7$ are all 120° (within one e.s.d.) and Be is essentially coplanar with its three O neighbours (again, within one e.s.d.). Although a range of Be–O distances is observed in each of $Rb_2Be_2Si_2O_7$, K_2BeO_2, and Y_2BeO_4, the mean Be–O bond lengths (1·553, 1·555, and 1·554 Å respectively) are in excellent agreement. $Ca_{12}Be_{17}O_{29}$ exhibits larger distances (1·58 Å) in its BeO_3 units.

Attempts to confirm the coordination of Be in $Rb_2Be_2Si_2O_7$ by IR spectroscopy proved fruitless. There was no evidence of bands attributable to Be–O vibrations, expected in the region 1000–2000 cm^{-1}.

The Si_2O_7 component of the beryllosilicate framework possesses, as its only element of point symmetry, a twofold axis which passes through the bridging O, parallel to **a**. There seems to be no standard configuration for Si_2O_7 groups and observed symmetries vary from structure to structure. For example, in hemimorphite, $Zn_4Si_2O_7(OH)_2.H_2O$ (Barclay & Cox, 1960), a twofold axis is also present but this is caused by the additional presence of two intersecting mirror planes, whereas in thortveitite, $Sc_2Si_2O_7$ (Zachariasen, 1930; Cruickshank, Lynton & Barclay, 1962), the Si_2O_7 adopts a staggered configuration with a centre of symmetry at the bridging O. The Si–O–Si angle is almost linear (171°) in $Rb_2Be_2Si_2O_7$, which is uncommon, (*cf.* 151° in hemimorphite) but not unknown (180° in thortveitite). Si–O bond lengths in $Rb_2Be_2Si_2O_7$ (Table 2) are comparable with those found previously. The Si-bridging O distance is somewhat larger than the other Si–O distances, as is normally the case.

The low electron densities at the O positions in the final Fourier map of $Rb_2Be_2Si_2O_7$ can be interpreted differently for the Si_2O_7 bridging O atom, O(3), and for the terminal O atoms, O(1), O(2), O(4). The bridging O atom, O(3), exhibits the highest temperature factor of all of the O atoms. Calvo (1965) observed the same phenomenon in the bridging O of the P_2O_7 group in β-$Zn_2P_2O_7$, but was unable to establish whether the effect indicated real vibration, or disorder.

The terminal O (and Si) atoms of the Si_2O_7 groups of $Rb_2Be_2Si_2O_7$ are the only atoms which are sensitive to the choice of space group between *I*2*mm* and *P*2*nn*,

---

* A list of (L1) structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32041 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 3. *Unit-cell data of* $Rb_2Be_2Si_2O_7$ *and related phases*

	a (Å)	b (Å)	c (Å)	$D_{X\text{-ray}}$ (g cm^{-3})	D_{obs} (g cm^{-3})
Rb_2BeSiO_4*	8·92	8·32	5·15	2·36	
$Rb_2Be_2Si_2O_7$	$8\cdot92_1$	$8\cdot32_1$	$5\cdot15_1$	3·11	3·2
Cs_2BeSiO_4	9·10	8·52	$5\cdot25_6$	2·99	
$Cs_2Be_2Si_2O_7$	$9\cdot12_4$	$8\cdot69_0$	$5\cdot26_8$	3·59	3·55
$Cs_2Be_3Si_3O_{10}$	9·08	8·80	$5\cdot25_2$	4·25	

* Some doubt is attached to the existence of this phase owing to the similarity between the X-ray powder diffraction patterns of Rb_2BeSiO_4 and $Rb_2Be_2Si_2O_7$.

which alters the site symmetry of the Si_2O_7 groups from *mm* to 2 and changes the Si_2O_7 configuration (in terms of the terminal O atoms) from fully eclipsed to slightly staggered [see (i) in Fig. 2]. It is tempting therefore, to regard the low electron densities associated with the terminal O atoms as evidence for disorder in the orientation of the distorted Si_2O_7 units. However, neither the final difference map, nor the improved *R* value for the structure in *P2nn* over both ordered and half-occupancy models in *I2mm* (see *Solution of the structure* above) supports this hypothesis. Crucial to the space group *P2nn*, especially in placing the Si and terminal O atoms, are the reflexions *hkl*, with $h + k + l$ odd. Since it was only possible to obtain crude estimates of the intensities of a very limited number of reflexions in this class, it is not perhaps surprising that the terminal O atoms are relatively ill-defined in the resulting electron density map.

Rb—O bond distances vary from 2·85 to 3·83 Å. Although some of the longer distances are greater than the closest Rb—Be (3·54 Å) and Rb—Si (3·49 Å) distances, it is felt justified to include these longer distances as bonds, since there is no obvious 'cut-off' or gap in Rb—O separations until 3·83 Å is reached.

The six-membered O rings, shown in Fig. 2, form windows to the Rb cages. The 'free diameter' of the windows is ~2·7 Å, approximately the diameter of an O atom. The beryllosilicate framework may be compared with the framework of an aluminosilicate zeolite. The lack of reproducibility of gas (air or nitrogen) pycnometry density determinations, and the persistently high densities obtained, are ascribed to the permeability of the $Rb_2Be_2Si_2O_7$ structure to molecular O or N, despite the presence of a Rb atom in each cage. Gas pycnometry was successfully applied to determining the density of $Cs_2Be_2Si_2O_7$ (see below), presumably because the larger Cs ion prevents sorption of O or N.

Other phases related to $Rb_2Be_2Si_2O_7$

In addition to the Rb beryllosilicates Rb_2BeSiO_4 and $Rb_2Be_2Si_2O_7$, three Cs beryllosilicates have been identified, Cs_2BeSiO_4, $Cs_2Be_2Si_2O_7$, $Cs_2Be_3Si_3O_{10}$ (or, possibly, $Cs_2Be_4Si_4O_{13}$). All five phases exhibit very similar X-ray powder diffraction patterns which can be indexed by analogy with $Rb_2Be_2Si_2O_7$ to give the unit cells given in Table 3.

There is little or no solid solution among these phases. For example a bulk composition corresponding to $Cs_4Be_3Si_3O_{11}$ yielded a mixture of Cs_2BeSiO_4 and $Cs_2Be_2Si_2O_7$.

The similarity in X-ray powder diffraction intensities in the patterns of all five phases implies the occurrence of the same approximately close-packed array of Rb or Cs atoms in each phase, arranged almost in $\cdots AAA \cdots$ fashion. From the relatively constant unit-cell dimensions through either series it is inferred that an increase in the beryllosilicate component results in an increasingly condensed framework, without the occurrence of vacant Rb or Cs ion sites. Detailed examination of the structural relations between these phases requires further single-crystal data.

We thank Mr B. G. Cooksley for collecting the intensity data and Dr L. S. Dent Glasser for advice. Mr N. Franklin carried out some of the early work as part of an Honours project. Computing facilities were kindly provided by the Computing Centre of Aberdeen University.

References

BARCLAY, G. A. & COX, E. G. (1960). *Z. Kristallogr.* **113**, 23–29.

CALVO, C. (1965). *Canad. J. Chem.* **43**, 1147–1153.

CRUICKSHANK, D. W. J., LYNTON, H. & BARCLAY, G. A. (1962). *Acta Cryst.* **15**, 491–498.

HARRIS, L. A. & YAKEL, H. L. (1966). *Acta Cryst.* B**20**, 295–301.

HARRIS, L. A. & YAKEL, H. L. (1967). *Acta Cryst.* B**22**, 354–360.

HOWIE, R. A. & WEST, A. R. (1976). *Nature, Lond.* **259**, 473.

International Tables for X-ray Crystallography (1968). Vol. III, 2nd ed., pp. 202–203. Birmingham: Kynoch Press.

KASTNER, P. & HOPPE, R. (1974). *Naturwissenschaften*, **61**, 79.

ZACHARIASEN, W. H. (1930). *Z. Kristallogr.* **73**, 1–6.