Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($Å^2$)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Be	3/4	0.2344 (13)	0	0.026 (4)
I	0.48630 (5)	−0.00410 (3)	0.078806 (11)	0.0144 (3)
O1*W*	0.7523 (5)	0.3161 (6)	0.03115 (12)	0.0320 (14)
O2*W*	0.6253 (5)	0.1548 (5)	0.00081 (11)	0.0306 (14)
O3	0.4194 (4)	0.0176 (4)	0.04080 (9)	0.0208 (11)
O4	0.5605 (4)	−0.1519 (4)	0.06973 (10)	0.0221 (11)
O5	0.3457 (4)	−0.0746 (4)	0.09501 (9)	0.0206 (11)
O6	0.6263 (4)	0.0805 (4)	0.06349 (10)	0.0238 (12)
O7	0.4277 (5)	0.1518 (4)	0.09268 (13)	0.0276 (13)
O8	0.5634 (5)	−0.0132 (5)	0.11893 (13)	0.0277 (13)

Table 2. *Selected geometric parameters (Å, °)*

IO_6 octahedron			
I—O3	1.799 (4)	I—O6	1.899 (4)
I—O4	1.844 (4)	I—O7	1.910 (5)
I—O5	1.850 (4)	I—O8	1.915 (5)
O3—I—O4	95.9 (2)	O5—I—O7	88.6 (2)
O3—I—O5	93.3 (2)	O6—I—O7	86.8 (2)
O4—I—O5	94.6 (2)	O3—I—O8	175.0 (2)
O3—I—O6	87.1 (2)	O4—I—O8	87.2 (2)
O4—I—O6	89.9 (2)	O5—I—O8	90.3 (2)
O5—I—O6	175.4 (2)	O6—I—O8	89.0 (2)
O3—I—O7	91.6 (2)	O7—I—O8	85.1 (2)
O4—I—O7	171.7 (2)		
BeO_4 tetrahedron			
Be—O1*W*	1.604 (9)	Be—O2*W*	1.613 (10)
Be—O1W^{iii}	1.604 (9)	Be—O2W^{iii}	1.613 (10)
O1*W*—Be—O1W^{iii}	112.5 (10)	O1*W*—Be—O2*W*	107.1 (3)
O1*W*—Be—O2W^{iii}	107.6 (2)	O1W^{iii}—Be—O2*W*	107.7 (2)
O1W^{iii}—Be—O2W^{iii}	107.1 (3)	O2W^{iii}—Be—O2*W*	115.0 (9)
Possible hydrogen bonds [$d(O\cdots O) < 3.3$ Å]			
O1$W\cdots O3^{i}$	2.638 (7)	$O5\cdots O8^{ix}$	3.157 (7)
O1$W\cdots O6$	3.226 (8)	$O6\cdots O5^{x}$	2.747 (6)
O1$W\cdots O4^{vi}$	2.648 (6)	$O6\cdots O7^{i}$	3.229 (7)
O2$W\cdots O3^{iv}$	2.636 (6)	$O7\cdots O7^{i}$	2.659 (9)
O2$W\cdots O6$	2.804 (6)	$O7\cdots O8^{xii}$	2.679 (8)
O2$W\cdots O3$	3.194 (7)	$O8\cdots O8^{vii}$	2.722 (9)
$O4\cdots O4^{v}$	2.513 (9)	$O8\cdots O4^{vii}$	3.276 (7)
$O5\cdots O5^{ix}$	2.588 (8)		

Symmetry codes: (i) $1-x, \frac{1}{2}-y, z$; (ii) $\frac{3}{2}-x, y-\frac{1}{2}, z$; (iii) $\frac{3}{2}-x, y, -z$; (iv) $1-x, -y, -z$; (v) $1-x, -\frac{1}{2}-y, z$; (vi) $\frac{3}{2}-x, \frac{1}{2}+y, z$; (vii) $\frac{3}{4}+y, x-\frac{3}{4}, \frac{1}{4}-z$; (viii) $\frac{3}{4}-y, x-\frac{1}{4}, \frac{1}{4}-z$; (ix) $\frac{1}{4}-y, \frac{1}{4}-x, \frac{1}{4}-z$; (x) $x+\frac{1}{2}, -y, z$; (xi) $x-\frac{1}{2}, -y, z$; (xii) $\frac{1}{4}+y, \frac{3}{4}-x, \frac{1}{4}-z$.

Polycrystalline samples of the compound were characterized by X-ray analysis using the Guinier technique with Cu $K\alpha$ radiation and quartz as internal standard. The unit-cell dimensions were calculated by least-squares methods (*LSUCR*: Least Squares Unit Cell Refinement, Programmbibliothek für die Chemie, HRZ Köln, Germany). The crystal studied was mounted in a sealed glass capillary. The space group was inferred from precession photographs of *hk*0, *hk*1, *hk*2, 0*kl*, 1*kl* and 2*kl* layers taken using a Buerger precession camera (Firma Huber, Rimsting) and powder data. The data were corrected for Lorentz and polarization effects. Empirical absorption corrections were applied *via* ψ scans of nine reflections in the range $3.86 < \theta < 24.72°$. The standard reflections were 10,0,0, 0,10,0, and 0,0,40. The positions of the H atoms were deduced from difference Fourier maps. H atoms were refined with a common isotropic displacement parameter (U_{iso} = 0.05 $Å^2$).

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *LSUCR*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX*. Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) and *SCHAKAL*92 (Keller, 1992).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: DU1154). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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$K_2Be_2Si_3O_9$, a Potassium Beryllium Silicate Framework Built from Edge-Sharing Be–O Tetrahedra and Three-Ring Silicate Tetrahedra

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Abstract

Dipotassium diberyllium trisilicon nonaoxide, $K_2Be_2Si_3O_9$, was synthesized at high temperature and high pressure and has a three-dimensional framework structure containing six-ring channels along the *b* axis. The framework is built from cyclic trimers of silicate tetrahedra and edge-sharing Be–O tetrahedra.

Comment

Zeolite structures based on two types of tetrahedral atoms which have a combined formal charge of +7 or

+8 are well known. These include aluminosilicates (+7), zincophosphates (+7) and aluminophosphates (+8). In comparison, there are relatively few zeolite structures based on tetrahedral atoms which have a combined formal charge of +6. The beryllo-silicate lovdarite is one of the zeolite minerals. It is hydrated and contains both Na^+ and K^+ cations (Merlino, 1981).

In the course of research focused on the preparation of beryllo-silicates, several high temperature and high pressure syntheses were carried out. In one of these, a new potassium beryllium silicate was synthesized, the structure of which is reported here.

The structure contains three unique Si^{4+} ions and two unique Be^{2+} ions (Fig. 1). The framework is built from cyclic silicate trimers and edge-sharing Be–O tetrahedra. Since the edge-sharing Be–O tetrahedra are located near the inversion centers at (0,0,0) and (1/2,1/2,1/2), each group consists of only one crystallographically unique Be cation and there are, therefore, two crystallographically independent Be–O clusters. However, there is only one unique three-ring Si_3O_9 unit.

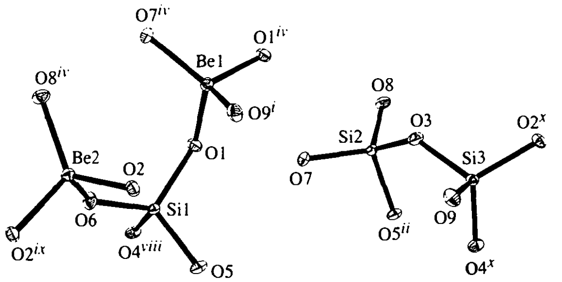

Fig. 1. The atomic labelling diagram showing the Si–O and Be–O tetrahedra. Displacement ellipsoids are drawn at the 50% probability level. Superscripts refer to the symmetry codes listed in Table 2.

Each silicate trimer is connected to six Be–O dimers (Fig. 2) and each Be–O dimer is connected to six silicate trimers (Fig. 3). Therefore, the Be:Si ratio is maintained at 2:3. The silicate trimer as a cluster is similar to an octahedral ion which is six-coordinated to other parts of the framework. The Be–O dimer is also six-coordinated to the rest of the framework since the bridging O atoms have trigonal coordination. Thus, the connectivity between the trimers and dimers is similar to a distorted corner-sharing octahedral framework.

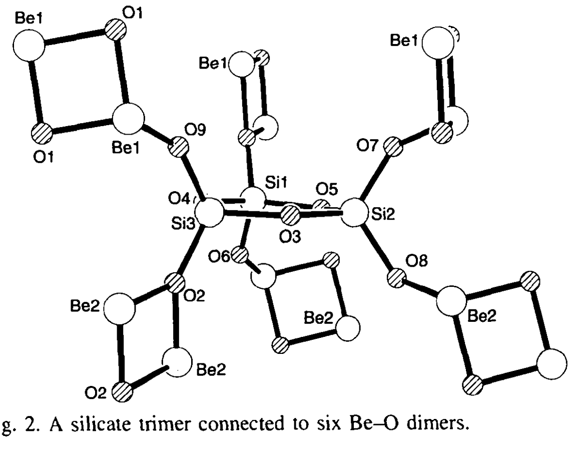

Fig. 2. A silicate trimer connected to six Be–O dimers.

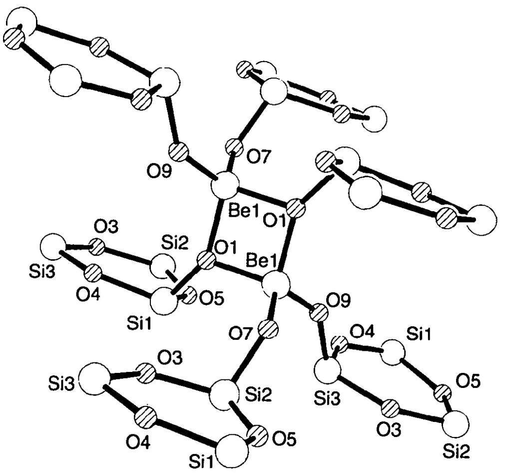

Fig. 3. A Be–O dimer connected to six silicate trimers. The cluster shown here has a center of inversion.

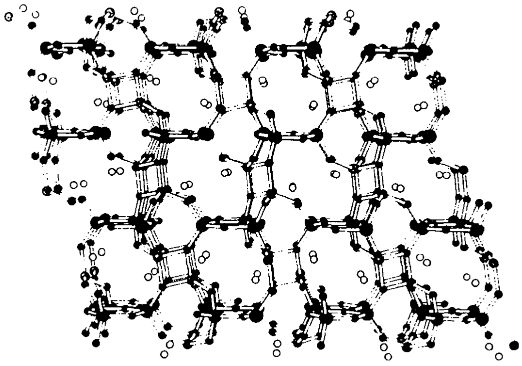

Fig. 4. The framework projected along the *b* axis showing the two types of six-ring channel. The large dark circles represent Si atoms, the small dark circles represent O atoms, the unconnected circles are K atoms and Be atoms are represented as lightly shaded medium-sized circles.

Two out of the nine crystallographically unique O atoms have trigonal coordination and are bridging O atoms in the Be–O dimer. The three-dimensional tetrahedral framework has two types of six-ring channels along the crystallographic *b* axis (Fig. 4). Two crystallographically unique K^+ cations are located in each channel near the wall (Fig. 4).

Experimental

To synthesize $K_2Be_2Si_3O_9$, 0.013 g of $Be(OH)_2$, 0.216 g of SiO_2 and 0.883 g of 4 *M* KOH were sealed into a gold tube 3 in long with a diameter of 1/4 in. The tube was heated in a Tempres apparatus (LECO corp.) to about 773 K and 35 000 p.s.i. (2.4×10^8 Pa). After a few hours, the bomb was slowly cooled to about 523 K over a period of 30 h and then to about 373 K for another 40 h. Further cooling to ambient conditions and recovery *via* standard filtration and washing techniques yielded 20 mg of small crystals suitable for the structure determination.

Crystal data

$K_2Be_2Si_3O_9$
$M_r = 324.49$
Monoclinic
$P2_1/c$
$a = 10.259\,(6)$ Å
$b = 6.940\,(4)$ Å
$c = 12.175\,(7)$ Å
$\beta = 106.764\,(7)^\circ$
$V = 830.0\,(8)$ Å^3
$Z = 4$
$D_x = 2.597$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 18 reflections
$\theta = 20$–22.5°
$\mu = 1.605$ mm^{-1}
$T = 293\,(2)$ K
Translucent plate
$0.17 \times 0.17 \times 0.067$ mm
Clear

Data collection

Huber four-circle diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{\min} = 0.678$, $T_{\max} = 0.862$
4926 measured reflections
2390 independent reflections

2201 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0641$
$\theta_{\max} = 29.93^\circ$
$h = 0 \rightarrow 14$
$k = -9 \rightarrow 9$
$l = -17 \rightarrow 16$
3 standard reflections monitored every 97 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0329$
$wR(F^2) = 0.0990$
$S = 1.206$
2390 reflections
145 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0189P)^2 + 1.1430P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.003$
$\Delta\rho_{\max} = 0.568$ e Å^{-3}
$\Delta\rho_{\min} = -0.768$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (*Å^2*)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
K1	0.45980 (5)	0.12625 (8)	0.64407 (4)	0.01702 (13)
K2	0.06300 (6)	0.60686 (9)	0.14762 (5)	0.02012 (14)
Be1	0.4288 (3)	0.5907 (4)	0.5422 (2)	0.0079 (5)
Be2	0.0650 (3)	0.5723 (4)	0.4424 (2)	0.0076 (5)
Si1	0.25741 (6)	0.31784 (9)	0.37323 (5)	0.00687 (13)
Si2	0.76207 (6)	0.11422 (8)	0.58064 (5)	0.00758 (13)
Si3	0.75823 (5)	−0.03130 (9)	0.81819 (5)	0.00707 (13)
O1	0.40109 (15)	0.4254 (3)	0.43324 (14)	0.0099 (3)
O2	0.10150 (15)	0.4809 (2)	0.57826 (14)	0.0092 (3)
O3	0.7646 (2)	0.1263 (2)	0.71782 (14)	0.0115 (3)
O4	0.2579 (2)	0.2544 (2)	0.74358 (13)	0.0100 (3)
O5	0.2406 (2)	0.1216 (2)	0.44478 (14)	0.0103 (3)
O6	0.1276 (2)	0.4505 (2)	0.35968 (14)	0.0108 (3)
O7	0.6224 (2)	0.1988 (2)	0.50017 (15)	0.0120 (3)
O8	0.8991 (2)	0.2022 (2)	0.56074 (15)	0.0112 (3)
O9	0.6239 (2)	0.0067 (3)	0.85612 (14)	0.0113 (3)

Table 2. *Selected geometric parameters* (*Å, °*)

K1—O9	2.771 (2)	Be1—O1$^{\rm iv}$	1.687 (3)
K1—O9$^{\rm i}$	2.776 (2)	Be1—O1	1.715 (3)
K1—O7	2.791 (2)	Be2—O6	1.585 (3)
K1—O5	2.792 (2)	Be2—O8$^{\rm iv}$	1.611 (3)
K1—O4	2.828 (2)	Be2—O2$^{\rm ix}$	1.694 (3)
K1—O7$^{\rm ii}$	2.834 (2)	Be2—O2	1.709 (3)
K1—O3	2.994 (2)	Si1—O6	1.588 (2)
K1—O1	3.220 (2)	Si1—O1	1.625 (2)
K2—O6	2.701 (2)	Si1—O5	1.652 (2)
K2—O8$^{\rm iii}$	2.742 (2)	Si1—O4$^{\rm viii}$	1.658 (2)
K2—O3$^{\rm iv}$	2.751 (2)	Si2—O7	1.596 (2)
K2—O8$^{\rm v}$	2.756 (2)	Si2—O8	1.614 (2)
K2—O5$^{\rm vi}$	2.993 (2)	Si2—O5$^{\rm ii}$	1.665 (2)
K2—O2$^{\rm vii}$	3.041 (2)	Si2—O3	1.665 (2)
K2—O6$^{\rm vi}$	3.069 (2)	Si3—O9	1.596 (2)
K2—O4$^{\rm viii}$	3.209 (2)	Si3—O2$^{\rm x}$	1.619 (2)
Be1—O7$^{\rm iv}$	1.587 (3)	Si3—O4$^{\rm x}$	1.653 (2)
Be1—O9$^{\rm i}$	1.597 (3)	Si3—O3	1.655 (2)
O7$^{\rm iv}$—Be1—O9$^{\rm i}$	115.7 (2)	O8—Si2—O3	111.69 (9)
O7$^{\rm iv}$—Be1—O1$^{\rm iv}$	110.3 (2)	O5$^{\rm ii}$—Si2—O3	103.35 (9)
O9$^{\rm i}$—Be1—O1$^{\rm iv}$	113.8 (2)	O9—Si3—O2$^{\rm x}$	114.43 (10)
O7$^{\rm iv}$—Be1—O1	113.5 (2)	O9—Si3—O4$^{\rm x}$	107.38 (9)
O9$^{\rm i}$—Be1—O1	109.7 (2)	O2$^{\rm x}$—Si3—O4$^{\rm x}$	110.56 (9)
O1$^{\rm iv}$—Be1—O1	91.44 (15)	O9—Si3—O3	108.98 (9)
O6—Be2—O8$^{\rm iv}$	111.5 (2)	O2$^{\rm x}$—Si3—O3	108.97 (9)
O6—Be2—O2$^{\rm ix}$	111.4 (2)	O4$^{\rm x}$—Si3—O3	106.19 (10)
O8$^{\rm iv}$—Be2—O2$^{\rm ix}$	115.8 (2)	Si1—O1—Be1$^{\rm iv}$	143.79 (15)
O6—Be2—O2	113.6 (2)	Si1—O1—Be1	125.56 (13)
O8$^{\rm iv}$—Be2—O2	113.3 (2)	Be1$^{\rm iv}$—O1—Be1	88.56 (15)
O2$^{\rm ix}$—Be2—O2	89.59 (15)	Si3$^{\rm i}$—O2—Be2$^{\rm ix}$	137.38 (14)
O6—Si1—O1	113.91 (10)	Si3$^{\rm i}$—O2—Be2	132.12 (13)
O6—Si1—O5	108.69 (9)	Be2$^{\rm ix}$—O2—Be2	90.41 (15)
O1—Si1—O5	111.13 (9)	Si3—O3—Si2	135.61 (11)
O6—Si1—O4$^{\rm viii}$	108.00 (9)	Si3$^{\rm i}$—O4—Si1$^{\rm xi}$	132.67 (11)
O1—Si1—O4$^{\rm viii}$	108.39 (9)	Si1—O5—Si2$^{\rm ii}$	135.31 (11)
O5—Si1—O4$^{\rm viii}$	106.40 (9)	Be2—O6—Si1	136.41 (14)
O7—Si2—O8	115.81 (10)	Be1$^{\rm iv}$—O7—Si2	133.81 (15)
O7—Si2—O5$^{\rm ii}$	106.49 (9)	Be2$^{\rm iv}$—O8—Si2	125.89 (14)
O8—Si2—O5$^{\rm ii}$	108.21 (9)	Si3—O9—Be1$^{\rm x}$	142.94 (14)
O7—Si2—O3	110.39 (10)		

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $1 - x, -y, 1 - z$; (iii) $x - 1, \frac{1}{2} - y, z - \frac{1}{2}$; (iv) $1 - x, 1 - y, 1 - z$; (v) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (vi) $-x, \frac{1}{2} + y, \frac{1}{2} - z$; (vii) $x, \frac{3}{2} - y, z - \frac{1}{2}$; (viii) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ix) $-x, 1 - y, 1 - z$; (x) $1 - x, y - \frac{1}{2}, \frac{3}{2} - z$; (xi) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

Data collection: *UCLA-CCP* (Strouse, 1991). Cell refinement: *UCLA-CCP*. Data reduction: *UCLA-CCP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ATOMS* (Dowty, 1994). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BR1147). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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