

tions. The intensity data were corrected for absorption, Lorentz, and polarization effects. Corrections for absorption effects were based on ψ scans of a few suitable reflections with χ values close to 90° . Max./min. transmission factors: 0.964/0.490. Systematic absences: $hk0$, $h+k=2n+1$; $0kl$, $l=2n+1$; hhl , $l=2n+1$. 290 unique reflections with $I > 3.0\sigma(I)$. Based on the statistical analysis of the intensity distribution, systematic extinctions, and the successful solution and refinement of the structure, the space group was determined to be $P4/ncc$ (No. 130). The structure was solved by direct methods and refined by full-matrix least squares based on F values. All of the atoms were refined with anisotropic temperature factors. At convergence $R = 0.0201$, $wR = 0.0167$, $w = [\sigma^2(F)]^{-1} [\sigma^2(F)]$ based on counting statistics, $(\Delta/\sigma)_{\max} = 0.021$, $S = 1.64$, $(\Delta\rho)_{\max} = 0.52$, $(\Delta\rho)_{\min} = -0.58 \text{ e } \text{Å}^{-3}$. Final atomic coordinates and thermal parameters are given in Table 1.* Atomic coordinates of gillespite $\text{BaFeSi}_4\text{O}_{10}$ and its lattice constants are also given in Table 1 for comparison. Selected bond distances and angles appear in Table 2. The linkage motif and the coordination numbers in $\text{BaCuSi}_4\text{O}_{10}$ are given in Table 3. The title compound is isotypic with $\text{BaFeSi}_4\text{O}_{10}$ (Pabst, 1943, 1958). Projections of the structure approximately along the a and c axes are shown in Fig. 1. The structure contains rings of four-connected SiO_4

tetrahedra. Each ring is connected to four others to form a silicate layer in the ab plane, as shown in Fig. 1(a). The Cu^{2+} and Ba^{2+} cations are located in the tunnels formed by rings of SiO_4 tetrahedra as shown in Fig. 1(b). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed on a DEC MicroVAX II computer system using the *SHELXTL-Plus* program (Sheldrick, 1990).

Related literature. The crystal structure of $\text{BaCuSi}_4\text{O}_{10}$ is isotypic with gillespite ($\text{BaFeSi}_4\text{O}_{10}$) which was reported by Pabst (1943, 1958). Both belong to the sheet silicate structure except that fourfold coordinated Fe^{2+} is substituted by fourfold coordinated Cu^{2+} .

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* Lists of anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54949 (4 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BX0572]

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Refinement of the Structure of Barium Copper Silicate $\text{BaCu}[\text{Si}_4\text{O}_{10}]$ at 300 K*

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Abstract. Barium copper tetrasilicate, $\text{BaCu}[\text{Si}_4\text{O}_{10}]$, $M_r = 473.2$, tetragonal, $P4/ncc$, $a = 7.447$ (1), $c = 16.138$ (2) Å, $V = 895.0$ (2) Å³, $Z = 4$, $D_x = 3.512 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 73.3 \text{ cm}^{-1}$, $T = 300 \text{ K}$, $F(000) = 884$, final $R = 0.030$,

$wR = 0.023$, for 625 independent reflections. The crystals have been obtained during a study of the chemical reactivity of the '1-2-3' type superconductors. The structure $\text{BaCu}[\text{Si}_4\text{O}_{10}]$ is of the $\text{BaFe}[\text{Si}_4\text{O}_{10}]$ structure type, reported by Pabst [*Acta Cryst.* (1959), **12**, 733–739]. The structure contains SiO_4 tetrahedra which share corners to form four-membered rings which in turn are linked *via* four

* See also preceding paper [Lin, Liao & Wang (1992). *Acta Cryst.* **C48**, 1297–1299].

Table 1. Final atomic coordinates and equivalent isotropic thermal parameters U_{eq} ($\times 10^4$), with *e.s.d.*'s in parentheses

$$U_{\text{eq}} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

Position	x	y	z	U_{eq} (\AA^2)
Ba	4(b)	0	0	100 (1)
Cu	4(c)	0	944 (1)	77 (3)
Si	16(g)	2681 (1)	1887 (1)	73 (4)
O(1)	8(f)	2261 (5)	2261 (5)	310 (8)
O(2)	16(g)	4736 (4)	2484 (5)	160 (7)
O(3)	16(g)	1367 (4)	2818 (4)	157 (7)

Table 2. Selected interatomic distances (\AA) and bond angles ($^\circ$) with *e.s.d.*'s in parentheses

Si—O(1)	1.595 (1)	Cu—O(3) 4 ×	1.918 (3)
Si—O(2) ⁱ	1.633 (3)	Ba—O(3) 4 ×	2.755 (3)
Si—O(2)	1.620 (4)	Ba—O(2) ^j 4 ×	2.900 (3)
Si—O(3)	1.581 (3)	Ba—O(3) ^k 4 ×	3.479 (3)
Si—O(1)—Si ⁱⁱ	178.3 (3)	O(1)—Si—O(2) ^j	109.2 (2)
O(1)—Si—O(2)	108.1 (2)	O(1)—Si—O(3)	115.5 (2)
O(2)—Si—O(3) ^j	105.5 (2)	O(2)—Si—O(3)	110.4 (2)

Bond angles in the [Si₄O₁₀]⁴⁻ ring
 O(2)—Si—O(2)^j 108.0 (2) Si—O(2)—Siⁱⁱⁱ 151.0 (2)

Symmetry code: (i) 0.5 - y, -0.5 + x, z, (ii) y, x, 0.5 - z;
 (iii) 0.5 + y, 0.5 - x, z.

corners to other, symmetrically equivalent rings to form Si₈O₂₀ layers. Ba and Cu cations are located between the layers. The Si—O distances range from 1.581 (3) to 1.633 (3) \AA . The copper cation is square coordinated by four O(3) atoms, with the Cu—O(3) distance 1.918 (3) \AA . The barium cation is coordinated by eight O atoms in a square antiprism, the distances are Ba—O(2) \times 4 = 2.900 (3), Ba—O(3) \times 4 = 2.755 (3) \AA .

Experimental. Crystals of BaCu[Si₄O₁₀] were obtained in the reaction of YBa₂Cu₃O_{7-x} with PbO and Na₂O₂ in the molar proportion 1:2:1. The mixture was pelletized, evacuated and sealed in a silica tube. The pellets were heated at 1100 K for 10 h and quenched in air. At the high temperature the mixture reacted with the tube walls and BaCu[Si₄O₁₀] was formed as a phase.

A dark-blue crystal limited by eight faces [the distances between the faces are equal: (110)—($\bar{1}\bar{1}0$) 0.230, (1 $\bar{1}0$)—($\bar{1}10$) 0.225, (010)—(0 $\bar{1}0$) 0.180, (001)—(00 $\bar{1}$) 0.085 mm, density not measured] was used for intensity data collection on a four-circle KM-4 diffractometer with graphite-monochromatized Mo K α radiation. Preliminary examination by rotation and Weissenberg photographs indicated space group *P4/ncc* which was used in the structure solution and refinement (origin at *4/ncn*). Lattice parameters were refined by the least-squares fit of 20 reflections in the range $15 \leq 2\theta \leq 25^\circ$. 2900 reflections (770 unique) were measured in the range $4 \leq 2\theta \leq 64^\circ$ ($\sin\theta/\lambda = 0.746 \text{ \AA}^{-1}$) using the ω - 2θ scan technique with scan

speed $0.05^\circ \text{ s}^{-1}$ and scan width 1.2° . The *hkl* range was $h 0 \rightarrow 10$, $k 0 \rightarrow 10$, $l -22 \rightarrow 22$. Two standard reflections (0 $\bar{2}4$, 400) were monitored every 50 reflections. They showed no significant intensity variations. The measured intensities were corrected for Lorentz and polarization effects. The absorption correction was calculated with the *SHELX76* program system (Sheldrick, 1976), the transmission factors were in the range 0.27749–0.53188. 625 independent reflections with $F > 2\sigma_F$ ($R_{\text{int}} = 0.030$) were used in subsequent calculations. The structure was solved by the Patterson method (Ba and Cu atoms) (*SHELXS86*, Sheldrick, 1990) and the remaining atoms were located from 4ρ maps. The structure was refined by the full-matrix least-squares method with anisotropic temperature factors (*SHELX76*, Sheldrick, 1976). An empirical secondary-extinction correction was applied according to the formula $F_{\text{corr}} = F(1 - 10^{-4}x F^2/\sin\theta)$, where x converged to 0.00176 (4). The function mini-

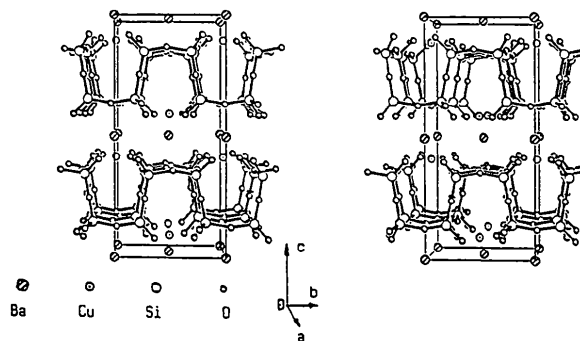


Fig. 1. A packing stereodiagram of molecules of the title compound.

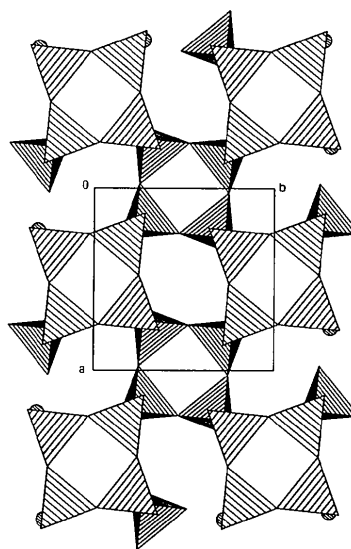


Fig. 2. The mode of linkage of SiO₄ tetrahedra.

mized was $\sum w(|F_o| - |F_c|)^2$ with $w = 3.3421/\sigma_F^2$. Final $R = 0.030$, $wR = 0.023$ for 39 refined parameters ($R = 0.052$, $wR = 0.024$ for all reflections), $(\Delta/\sigma)_{\max} < 0.002$. Residual electron density in final $\Delta\rho$ map was within -0.54 to $0.45 \text{ e } \text{\AA}^{-3}$. Scattering factors including corrections for anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Figures were drawn with program *Graphics* from the *SHELXTL* program system (Sheldrick, 1990). All calculations were performed with an IBM PC/AT.

Final atomic parameters and isotropic temperature factors are reported in Table 1.* Selected interatomic distances and bond angles are given in Table 2. A stereodrawing of the unit-cell contents is shown in Fig. 1. Fig. 2 shows the layer consisting of two planes of the four-membered rings, linked by bridging O(1) atoms.

Related literature. The title crystal is the third barium copper silicate obtained during our studies on the chemical reactivity of the '1-2-3' type superconductors. Results on the synthesis and crystal struc-

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54946 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0258]

ture of $\text{BaCu}_2[\text{Si}_2\text{O}_7]$ and $\text{Ba}_2\text{Cu}_2[\text{Si}_4\text{O}_{12}]$ have been published previously (Janczak, Kubiak & Głowiak, 1990; Janczak & Kubiak, 1992). The structure $\text{BaCu}[\text{Si}_4\text{O}_{12}]$ is isostructural with $\text{BaFe}[\text{Si}_4\text{O}_{10}]$, $\text{CaCu}[\text{Si}_4\text{O}_{10}]$ and $\text{SrCu}[\text{Si}_4\text{O}_{10}]$, reported by Pabst (1954, 1959). The compound $\text{CaCr}[\text{Si}_4\text{O}_{10}]$ is also apparently isostructural, Belsky (1984). The $\text{KCa}_4[\text{Si}_8\text{O}_{20}]$ structure contains $[\text{Si}_4\text{O}_{10}]^{4-}$ groups as in $\text{BaCu}[\text{Si}_4\text{O}_{10}]$ (Stähl, Kvick & Ghose, 1987). The Si—O distances in $\text{BaCu}[\text{Si}_4\text{O}_{10}]$ are comparable with those in other tetrasilicates.

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Structure of KErTe_2

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Abstract. Potassium erbium ditelluride, KErTe_2 , $M_r = 461.56$, trigonal, $R\bar{3}m$, $a = 4.410$ (2), $c = 24.27$ (2) \AA , $V = 408.8$ (8) \AA^3 , $Z = 3$, $D_x = 5.626 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha_1) = 0.7093 \text{ \AA}$, $\mu = 267 \text{ cm}^{-1}$, $F(000) = 567.3$ (including anomalous dispersion), $T = 111 \text{ K}$, $R(F) = 0.034$ for 202 unique observations with $F_o^2 > 3\sigma(F_o^2)$. KErTe_2 adopts the $\alpha\text{-NaFeO}_2$ structure, an NaCl superstructure where the cations are ordered in alternating layers between cubic close-packed anions.

Experimental. KErTe_2 was prepared through the use of a flux consisting of K_2Te_3 and Te in a ratio of 1:3.

K_2Te_3 was prepared by the reaction of K (Alfa, 99%) and Te (Aldrich, 99.8%) in the stoichiometric ratio of 2:3 in an evacuated quartz tube at 923 K for three days. In a dry box under an Ar atmosphere, 0.249 g K_2Te_3 , 0.045 g Er (REacton, 99.9%), and 0.206 g Te were ground together and then loaded into a quartz tube. The tube was evacuated, heated at 873 K for 4 d, and then ramped to 1173 K to heat for 4 d. The tube was cooled at the rate of 3 K h^{-1} to 648 K and then to room temperature at 99 K h^{-1} . Black air-stable hexagonal plates were obtained. The presence of the three elements K, Er and Te was confirmed by EDAX (energy-dispersive analysis by X-rays) measurements with a Hitachi S570 scanning electron microscope.

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