Table 4. *Bond angles* (°)

C(1)—S——C(6)	87·6 (4)
S——C(1)-O(1)	107·0 (3)
C(1)—O(1)-C(9)	110·8 (6)
O(1)—C(9)-C(6)	115·3 (5)
S——C(6)-C(9)	111·9 (4)
S——C(1)-C(2)	112·4 (4)
S——C(1)-C(4)	106·5 (3)
S——C(6)-C(7)	115·5 (4)
C(2)—C(1)-O(1)	108·7 (6)
C(2)—C(1)-C(4)	112·1 (5)
C(1)—C(4)-O(3)	117·7 (6)
C(1)—C(4)-C(5)	118·1 (6)
C(1)—C(2)-O(2)	121·2 (6)
C(1)—C(2)-C(3)	116·0 (7)
C(4)—C(1)-O(1)	108·7 (6)
C(3)—C(2)-O(2)	122·9 (7)
C(5)—C(4)-O(3)	124·1 (6)
C(7)—C(6)-C(9)	132·5 (7)
O(4)—C(7)-C(8)	120·2 (6)
C(6)—C(7)-O(4)	118·5 (6)
C(6)—C(7)-C(8)	121·3 (7)
C(6)—C(9)-C(10)	134·0 (6)
C(10)-C(9)-O(1)	110·7 (7)

as opposed to a theoretical 540° for a planar model. The non-planarity consists mainly of a displacement of C(1) from the mean plane of the other four atoms.

We thank Professor Taylor for giving us this interesting compound.

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SHORT COMMUNICATIONS

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The crystal structure of Ba$_2$SiS$_4$.

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Crystals of Ba$_2$SiS$_4$ are orthorhombic, space group *Pnma*, with four formula equivalents in a unit cell of dimensions $a=8{\cdot}93$, $b=6{\cdot}78$ and $c=12{\cdot}01$ Å. The structure was determined by standard Patterson and Fourier methods and is of the K$_2$SO$_4$ type. Intensity data were collected on a quarter-circle manual diffractometer. An R value of 0·061 was obtained for 695 observed reflections after least-squares refinement.

A crystallographic investigation of Ba$_2$SiS$_4$ was undertaken in an effort to confirm Susa & Steinfink's (1971) prediction on the basis of the ratio of atomic radii, $r_{\text{Si}}/r_{\text{Ba}}$, and a force constant, that its structure should correspond to that of K$_2$SO$_4$.

Experimental

Amber crystals of Ba$_2$SiS$_4$ were prepared in this laboratory by heating a stoichiometric mixture of barium sulfide, silicon, and sulfur at 1100° for 26h. Cooling was accomplished stepwise over approximately three days. The sample charges were placed in closed graphite crucibles and sealed in evacuated vycor tubes for firing.

A thin elongated hexagonal platelet (approximately $0{\cdot}20 \times 0{\cdot}10 \times 0{\cdot}01$ mm) was selected for X-ray examination. Diffraction symmetry and systematic extinctions ($0kl$, $k+l=2n+1$; $hk0$, $h=2n+1$) correspond to the orthorhombic space group *Pnma*. A least-squares refinement based upon 44 diffraction maxima measured on a General Electric XRD-5 manual diffractometer at 2θ values between 31 and 50° with Mo $K\alpha_1(\lambda=0{\cdot}7135\text{A})$ and Mo $K\alpha_2(\lambda=0{\cdot}7093$ Å) radiation, yielded the lattice parameters: $a=8{\cdot}9304\pm0{\cdot}0009$, $b=6{\cdot}7821\pm0{\cdot}0004$, $c=12{\cdot}0106\pm0{\cdot}0008$ Å. $V=727{\cdot}4$ Å^3; $Z=4$; $D_{\text{calc}}=3{\cdot}936$ g cm^{-3}.

Crystal structure determination

Diffraction intensities were measured on the XRD-5 diffractometer using Zr-filtered Mo $K\alpha$ ($\lambda=0{\cdot}7107$) radiation and a stationary-crystal stationary-counter technique. Of the 905 non-systematically extinct reflections measured, 695 were regarded as observed on the basis that the peak count exceeded the background count by at least 6 counts in 20s; that is, approximately twice the standard deviation. Extinction corrections were found to be unnecessary, but Lorentz, polarization, and absorption corrections were

Table 1. *Atomic parameters* (× 10^4)

The estimated standard deviations in parentheses apply to the last significant digit.
The temperature factor is: $\exp\left[-(B_{11}h^2+B_{22}k^2+B_{33}l^2+2B_{12}hk+2B_{13}hl+2B_{23}kl)\right]$.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Ba(1)	4799 (2)	$\frac{1}{4}$	1716 (2)	37 (2)	91 (4)	18 (1)	0	1 (2)	0
Ba(2)	1348 (2)	$\frac{1}{4}$	9067 (2)	48 (3)	88 (5)	53 (2)	0	12 (2)	0
Si	2322 (9)	$\frac{1}{4}$	5754 (7)	21 (10)	22 (16)	12 (6)	0	−2 (6)	0
S(1)	4985 (9)	$\frac{1}{4}$	9034 (7)	16 (9)	113 (19)	23 (6)	0	8 (6)	0
S(2)	1758 (7)	4958 (9)	1482 (5)	57 (8)	70 (11)	42 (5)	6 (8)	19 (5)	21 (6)
S(3)	3087 (10)	$\frac{1}{4}$	4099 (7)	35 (10)	142 (21)	19 (6)	0	4 (7)	0

made. The structure was solved by standard Patterson and Fourier methods. Final full-matrix least-squares refinement based upon 40 independent parameters, including a scale factor, 13 positional parameters and 26 anisotropic thermal parameters, yielded a standard residual $R=0{\cdot}061$. The unobserved reflections were excluded from the refinement but a structure-factor calculation based on the final atomic parameters and all 905 measured reflections yielded $R=0{\cdot}090$. Structure factors of unobserved reflections were calculated by assuming the intensity to be 3 counts above background and applying the appropriate corrections.*

Discussion

The structure corresponds to that of K_2SO_4 as predicted by Susa & Steinfink (1971). The positional parameters correlate closely with those of other K_2SO_4-type structures (Wyckoff, 1965). Table 1 is a listing of atomic positional coordinates and thermal parameters.

Table 2 is a list of selected bond distances and angles. Both barium atoms are coordinated to distorted trigonal prisms of sulfur atoms, the lateral faces of which are capped by additional sulfur atoms. Ba(2) has additional sulfur atoms capping the triangular end faces as well. Trigonal prismatic coordination between barium atoms and surrounding sulfur atoms is common, as is the capping of one or more of the lateral faces (Hong & Steinfink, 1972). The present instance of elevenfold coordination about Ba(2) is somewhat unusual; however, three of the sulfur atoms are found at extraordinary distances (> 3·9 Å *vs.* an average of 3·33 Å for the other Ba–S distances in the structure) and therefore correlate with substantially weaker bonds.

The silicon atom is tetrahedrally coordinated by sulfur atoms, all Si–S distances being 2·10 Å. The tetrahedra angles S–Si–S range from 105·5 to 115·9°. The tetrahedra are completely isolated from each other in the structure.

Table 2. *Interatomic distances and angles*

The estimated standard deviations in parentheses apply to the last significant digit.

Ba(1)–S(1)	3·225 (12) Å	Ba(2)–2S(2)	3·332 (11) Å
Ba(1)–2S(2)	3·199 (11)	Ba(2)–2S(2)	3·365 (11)
Ba(1)–2S(2)	3·245 (11)	Ba(2)–2S(2)	3·909 (11)
Ba(1)–S(3)	3·095 (12)	Ba(2)–S(1)	3·248 (12)
Ba(1)–2S(1)	3·514 (12)	Ba(2)–S(1)	3·918 (12)
Ba(1)–S(3)	3·246 (12)	Ba(2)–S(3)	3·651 (13)
Ba(1)–Ba(2)	4·429 (4)	Ba(2)–2S(3)	3·429 (13)
Ba(1)–2Ba(1)	4·846 (4)	Ba(2)–2Ba(2)	4·724 (5)
Si–S(1)	2·103 (16)		
Si–2S(2)	2·100 (16)		
Si–S(3)	2·101 (17)		
Si–Ba(2)	3·602 (12)		
Si–Ba(1)	3·725 (12)		
Si–2Si	5·654 (17)		

S(2)–Si–S(2)	110·3 (6)°
2[S(1)–Si–S(2)]	109·7 (6)
2[S(2)–Si–S(3)]	105·5 (6)
S(1)–Si–S(3)	115·9 (6)

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* A table of observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30253 (6 pp.). Copies maybe obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.