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THE CRYSTAL STRUCTURE OF SARABAUITE $CaSb_{10}O_{10}S_6$, A NEW OXIDE SULFIDE MINERAL

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Sarabauite is monoclinic: a=25.33(2), b=5.655(3), c=16.88(1) Å and β=117.51(6)° and C2/c. Z=4 and ρ_c=4.99 g/cm^3. The Sb is essentially coordinated by three atoms of oxygen and/or sulfur. SbO_3 pyramids of the Sb(1), Sb(2) and Sb(3) share corners forming a slab parallel to (100). The four membered ring consisting of $Sb(S,O)_3$ pyramids of the Sb(4) and Sb(5) links to the slab through the O(2). The Ca is coordinated by eight oxygen atoms in the slab.

Sarabauite, $CaSb_{10}O_{10}S_6$, is a new mineral from Sarabau mine, Sarawak, Malaysia[1] and the second oxide sulfide mineral subsequent to kermesite,[2] Sb_2S_2O. It is monoclinic. The cell dimensions were determined with the least-squares refinement using the 2θ values of 32 reflections measured on a Rigaku four-circle diffractometer. They are a=25.33(2), b=5.655(3), c=16.88(1) Å and β=117.51(6)°, with Z=4. MoKα$_1$ radiation (0.709258 Å) was used. The space group was determined to be C2/c by the N(z) test. Intensities were measured using a single crystal of 0.09x0.1x0.12 mm in size on the same instrument with Zr-filtered MoKα radiation. Symmetry-independent 2519 reflections were obtained within the limit of 2θ≤55°. Lorentz and polarization corrections were applied.

Three-dimensional Patterson synthesis was carried out using all the reflection data. Positions of the antimony atoms were determined by the minimum function method. The calcium, sulfur and oxygen atoms were found in the subsequent Fourier and difference Fourier syntheses. The coordinates and isotropic temperature factors were refined by the full-matrix least squares method. The R values were 0.105 and 0.084 for all and non-zero reflections, respectively. The atomic coordinates are given in Table 1. The scattering factors for neutral atoms were taken from International Tables for X-ray Crystallography.[3] Selected metal-anion bond lengths less than 3 Å are in Table 2.

The structure projected along the b axis is shown in Fig. 1. The three shortest Sb-O and/or Sb-S bonds of each antimony atom are drawn. The Sb(1), Sb(2) and Sb(3), which are coordinated only by oxygen atoms, share oxygens to make a slab parallel to (100). The four membered ring consisting of the $Sb(S,O)_3$ pyramids of the Sb(4) and Sb(5) links to the slab through the O(2). The S(3) is coordinated by an Sb(5), resulting in a large thermal motion compared with other sulfur and oxygen atoms(Table 1). The Ca is located at the center of oxygen polyhedron in the slab with the coordination number of eight.

Further refinement with extinction correction is now in progress.

Table 1. Atomic coordinates and isotropic temperature factors.

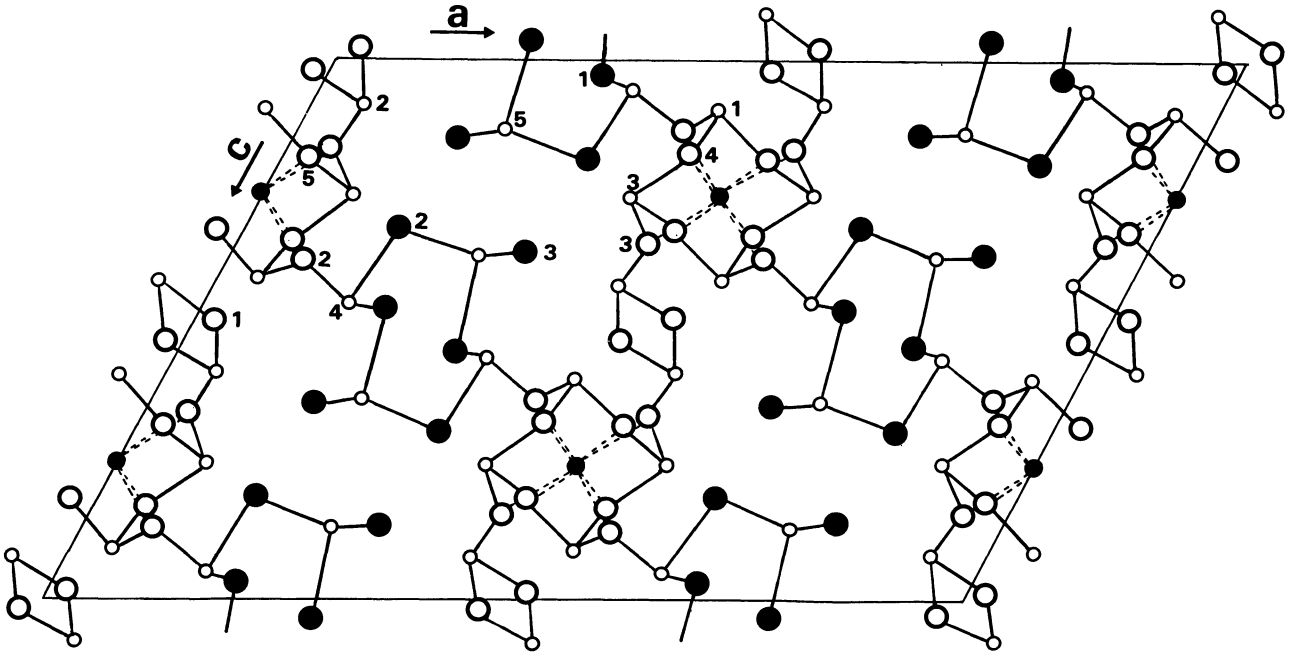
Atom	x	y	z	B/Å^2
Sb(1)	0.4517(1)	0.4910(3)	0.0881(1)	0.85
Sb(2)	0.0605(1)	0.4400(3)	0.0843(1)	0.95
Sb(3)	0.4019(1)	0.3905(3)	0.2478(1)	0.77
Sb(4)	0.1644(1)	0.3857(3)	0.4519(1)	0.99
Sb(5)	0.2275(1)	0.1009(3)	0.1343(1)	1.49
Ca	0.0000	0.4460(11)	0.2500	0.72
S(1)	0.2956(3)	0.4800(11)	0.0260(4)	1.23
S(2)	0.1723(3)	0.4286(11)	0.3109(4)	1.28
S(3)	0.3244(4)	0.2623(17)	0.3567(6)	3.11
O(1)	0.0237(8)	0.3284(35)	0.4827(13)	1.70
O(2)	0.0838(7)	0.2467(30)	0.3727(11)	0.89
O(3)	0.4540(7)	0.1461(31)	0.3352(11)	1.13
O(4)	0.4381(7)	0.2788(31)	0.1740(11)	1.06
O(5)	0.0313(7)	0.1178(29)	0.1793(11)	0.87

Table 2. Selected bond lengths (Å) in $CaSb_{10}O_{10}S_6$.

Sb(1) - O(1)	2.479(22)	Sb(2) - O(1)	2.000(18)	Sb(3) - O(3)	2.008(16)
- O(2)	1.971(19)	- O(1')	2.012(19)	- O(4)	1.960(22)
- O(3)	2.886(17)	- O(3)	1.950(20)	- O(5)	2.026(15)
- O(4)	2.031(20)	- O(5)	2.744(19)	Ca - O(2)	2.450(14)x2
- O(5)	2.021(14)	Sb(5) - S(1)	2.531(7)	- O(3)	2.501(22)x2
Sb(4) - O(2)	2.013(15)	- S(2)	2.465(7)	- O(4)	2.406(17)x2
- S(1)	2.466(7)	- S(3)	2.367(11)	- O(5)	2.525(20)x2
- S(2)	2.490(8)				

Fig. 1. The structure of $CaSb_{10}O_{10}S_6$ projected on (010).

● Ca ○ Sb ○ O ● S



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