Crystal structure of potassium ytterbium(III) tetrathiosilicate, $KYbSiS_4$

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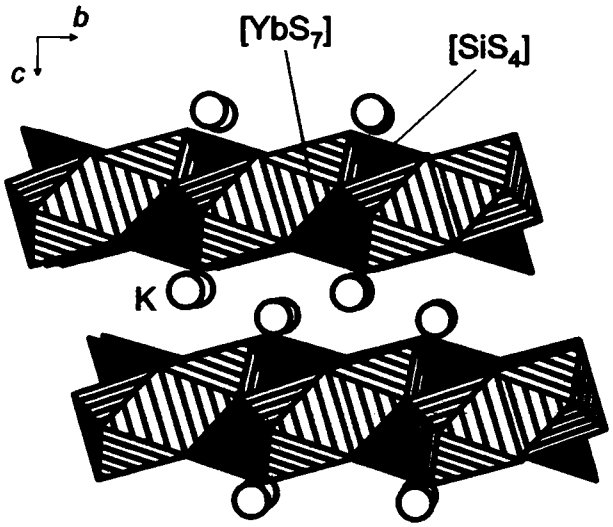

Abstract

KS_4SiYb, monoclinic, $P12_11$ (no. 4), $a = 6.316(5)$ Å, $b = 6.557(5)$ Å, $c = 8.571(7)$ Å, $\beta = 108.09(1)°$, $V = 337.4$ Å^3, $Z = 2$, $R_{gt}(F) = 0.025$, $wR_{ref}(F^2) = 0.059$, $T = 298$ K.

Source of material

Crystals of $KYbSiS_4$ were formed from a molten chalcogenide flux reaction of 83.8 mg Yb, 12.8 mg Si, 30.8 mg S, and 68.9 mg K_2S_2. These reactants were placed in a fused silica ampoule in an inert atmosphere glovebox. The ampoule was sealed under vacuum, and heated to 750 °C at a rate of 35 K/h. After 150 hours of annealing, the ampoule was cooled at 4 K/h to room temperature. Orange plates of $KYbSiS_4$ were obtained after the product was washed with dimethylformamide to dissolve remaining flux.

Discussion

$KYbSiS_4$ is isostructural to the previously reported quaternary compounds $KLaGeQ_4$ (Q = S, Se) [1], $KEuSiS_4$ [2], $KSmGeS_4$ [3], and KTbGeS4 [4]. The structure contains tetrathiosilicate(IV) anions $[SiS_4]^{4-}$, and Yb1 is coordinated to seven sulfur atoms from four different $[SiSe_4]^{4-}$ anions in a mono-capped trigonal prismatic fashion. $KYbSiS_4$ is a two-dimensional structure with $^2_\infty\{[YbSiS_4]^-\}$ layers separated by potassium cations and lying parallel to (001).

Table 1. Data collection and handling.

Crystal:	orange plate, size 0.05 × 0.40 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	157.58 cm^{-1}
Diffractometer, scan mode:	Bruker AXS SMART CCD, φ/ω
$2\theta_{max}$:	46.42°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2025, 960
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 914
$N(param)_{refined}$:	65
Programs:	SHELXS-97 [5], SHELXL-97 [6], X-Seed-A [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
K(1)	2a	0.2699(5)	0.188(1)	0.0642(3)	0.038(1)	0.035(3)	0.019(1)	0.004(2)	0.010(1)	0.004(2)
Yb(1)	2a	0.23071(6)	0.16631(9)	0.54946(5)	0.0117(2)	0.0142(2)	0.0141(3)	−0.0003(3)	0.0048(2)	0.0002(4)
Si(1)	2a	0.2179(5)	−0.2966(6)	0.3260(4)	0.012(1)	0.013(3)	0.014(1)	−0.001(2)	0.005(1)	0.000(1)
S(1)	2a	0.0175(6)	0.4392(5)	0.2998(5)	0.015(2)	0.013(2)	0.017(2)	−0.001(2)	0.003(2)	0.000(2)
S(2)	2a	−0.0249(6)	−0.0616(5)	0.2815(5)	0.016(2)	0.009(2)	0.016(2)	0.001(1)	0.005(2)	0.000(2)
S(3)	2a	0.5833(4)	0.2025(7)	0.8298(3)	0.013(1)	0.030(3)	0.012(1)	0.000(1)	0.005(1)	−0.002(1)
S(4)	2a	0.4254(5)	−0.2408(4)	0.5690(4)	0.015(2)	0.017(1)	0.014(2)	0.001(1)	0.006(1)	−0.002(1)

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References

1. Wu, P.; Ibers, J. A.: Synthesis and structures of the quaternary chalcogenides of the type $KLnMQ_4$ (Ln = La, Nd, Gd, Y; M = Si, Ge; Q = S, Se). J. Solid State Chem. **107** (1993) 347-355.
2. Evenson, C. R.; Dorhout, P. K.: Synthesis and Characterization of Four New Europium Group XIV Chalcogenides: K_2EuTSe_5 and $KEuTS_4$ (T = Si, Ge). Inorg. Chem. **40** (2001) 2409-2414.
3. Martin, B. R.; Dorhout, P. K.: Molten Flux Synthesis of an Analogous Series of Layered Alkali Samarium Selenogermanate Compounds. Inorg. Chem. **43** (2004) 385-391.
4. Chan, B. C.; Dorhout, P. K.: Crystal structures of potassium terbium tetrasulfidogermanate, $KTbGeS_4$, and potassium praseodymium tetraselenidogermanate, $KPrGeSe_4$. Z. Kristallogr. NCS **220** (2005) 7-8.
5. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
6. Sheldrick, G. M.: SHELXS-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
7. Barbour, L. J.: X-Seed-A. Software tool for supramolecular crystallography. J. Supramol. Chem. **1** (2001) 189-191.

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