Crystal structure of decapotassium hexaarsenidodistannate(IV), $K_{10}[Sn_2As_6]$

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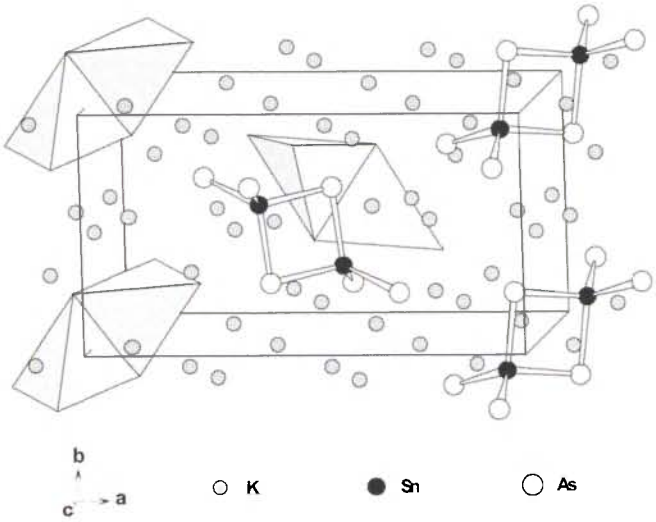

Abstract

$As_6K_{10}Sn_2$, monoclinic, $P12_1/n1$ (No. 14), $a = 15.1959(9)$ Å, $b = 8.2988(7)$ Å, $c = 9.219(1)$ Å, $\beta = 90.00(1)°$, $V = 1162.6$ Å^3, $Z = 2$, $R_{gt}(F) = 0.041$, $wR_{ref}(F^2) = 0.135$, $T = 293$ K.

Source of material

The compound was prepared by heating stoichiometric amounts of the elements under argon in corundum crucibles enclosed in steel containers to 923 K, tempering for one hour and cooling down to room temperature. The crystals forming dark metallic plates are very sensitive against moist air.

Discussion

$K_{10}[Sn_2As_6]$ crystallizes in the $Na_{10}[Si_2P_6]$ structure type [1]. The structure can be described starting from a strongly distorted hexagonal close packing of As^{3-} ions. All octahedral voids and 1/3 of the tetrahedral voids are occupied by K^+ cations. Sn occupies 1/6 of the tetrahedral voids in such a way that moieties of two edge sharing tetrahedra $[Sn_2As_6]^{10-}$ result being separated by K^+ cations. This connection via a common edge leads to a four-membered ring Sn_2As_2 with endocyclic bond lengths Sn—As of 2.703(1) Å and 2.712(1) Å, and an angle As–Sn–As of 92.49(3)°. The exocyclic bond lengths Sn—As are 2.630(1) Å and 2.636(1) Å with a bond angle As–Sn–As of 114.62(3)°, while the bond angles As–Sn–As between exo- and endocyclic As range between 110.57(3)° and 113.67(3)°. Longer known homologue representatives of this structure type are the compounds $K_{10}[Sn_2Sb_6]$ [2] and $K_{10}[Sn_2Bi_6]$ [3].

Table 1. Data collection and handling.

Crystal:	dark metallic, plate, size 0.08 × 0.15 × 0.17 mm
Wavelength:	Mo K_α radiation (0.71070 Å)
μ:	123.93 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	60.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7597, 3394
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2485
$N(param)_{refined}$:	82
Programs:	SHELXL-97 [4], DIAMOND [5]

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)	4e	0.7403(1)	0.1652(2)	0.4220(2)	0.0326(9)	0.0414(9)	0.031(1)	0.0058(7)	−0.0021(7)	0.0013(8)
K(2)	4e	0.4209(1)	0.1765(3)	0.4186(2)	0.033(1)	0.058(1)	0.032(1)	−0.0041(9)	−0.0036(8)	−0.0027(9)
K(3)	4e	0.0909(1)	0.0030(2)	0.8077(2)	0.036(1)	0.0371(9)	0.039(1)	−0.0087(8)	−0.0030(8)	0.0064(8)
K(4)	4e	0.0770(2)	0.5176(2)	0.6620(3)	0.053(1)	0.0325(9)	0.051(1)	0.0113(8)	0.013(1)	0.0089(9)
K(5)	4e	0.2590(1)	0.9905(2)	0.1940(2)	0.045(1)	0.0396(9)	0.035(1)	0.0150(8)	−0.0065(8)	−0.0052(8)
Sn(1)	4e	0.08569(3)	0.13925(5)	0.42518(5)	0.0191(2)	0.0195(2)	0.0205(3)	−0.0001(2)	−0.0009(2)	0.0006(2)
As(1)	4e	0.22483(5)	0.22685(9)	0.57545(9)	0.0224(4)	0.0278(4)	0.0270(5)	−0.0026(3)	−0.0031(3)	−0.0006(3)
As(2)	4e	0.07977(5)	0.25521(9)	0.15917(9)	0.0238(4)	0.0288(4)	0.0235(4)	−0.0011(3)	−0.0010(3)	0.0044(3)
As(3)	4e	0.93561(5)	0.18522(9)	0.57705(9)	0.0224(4)	0.0195(3)	0.0289(4)	0.0013(3)	0.0024(3)	−0.0007(3)

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