Crystal structure of hexapotassium niobium triarsenide oxide, $K_6[NbAs_3O]$

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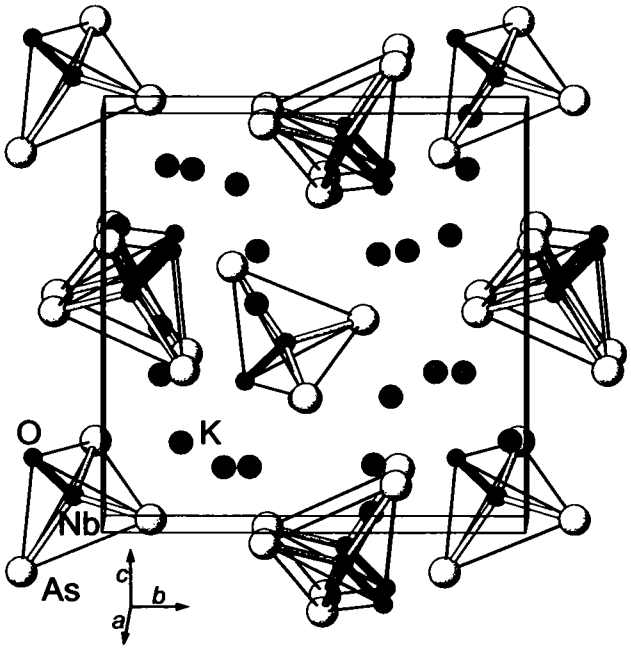
Received June 7, 2002, accepted and available on-line August 6, 2002; CSD-No. 409630



Abstract

As_3K_6NbO, cubic, $P2_13$ (No. 198), $a = 11.0409(5)$ Å, $V = 1345.9$ Å^3, $Z = 4$, $R_{gt}(F) = 0.040$, $wR_{ref}(F^2) = 0.105$, $T = 293$ K.

Source of material

K_6NbAs_3O was obtained as a minor component in the synthesis of K_7NbAs_4 at 870 K [1]. The compound is very sensitive to air and moisture, and turns immediately to a brown powder when exposed to air.

Experimental details

The absolute configuration with respect to the polarity of the space group was proven by changing the sign of the *hkl* indices. The $wR(F^2)$ values for both orientations were 0.105 and 0.196. The first one represents the absolute structure of the crystal studied.

Discussion

$K_6[NbAs_3O]$ crystallizes in a new *cP*44 structure type. Discrete monooxo-triarsenido-niobate(V) anions $[NbAs_3O]^{6-}$ with C_{3v} symmetry are arranged in almost fcc packing with the polar niobium oxygen bond oriented along the four threefold axes. Bond lengths are d(Nb—As) = 2.511(1) Å (3×), and d(Nb—O) = 1.829(6) Å (1×) corresponding with the covalent radii sums, and in agreement with the corresponding units of Ba_3NbAs_3O [2], and Eu_3TaAs_3O [3].

Table 1. Data collection and handling.

Crystal:	block with black metallic lustre, size 0.25 × 0.30 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	100.22 cm^{-1}
Diffractometer, scan mode:	Stoe STADI4, $2\theta/\omega$
$2\theta_{max}$:	59.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1496, 1315
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1297
$N(param)_{refined}$:	35
Programs:	SHELXS-97 [4], SHELXL-97 [5], ATOMS [6]

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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}
Nb	4*a*	0.43019(4)	x	x	0.0232(2)	U_{11}	U_{11}	0.0001(1)	U_{12}	U_{12}
As	12*b*	0.30574(5)	0.61373(5)	0.48073(5)	0.0340(3)	0.0273(3)	0.0311(3)	0.0050(2)	0.0017(2)	–0.0024(2)
K(1)	4*a*	0.1822(2)	x	x	0.0480(6)	U_{11}	U_{11}	0.0147(7)	U_{12}	U_{12}
K(2)	4*a*	0.6511(2)	x	x	0.0444(5)	U_{11}	U_{11}	0.0048(5)	U_{12}	U_{12}
K(3)	4*a*	0.8625(2)	x	x	0.0538(6)	U_{11}	U_{11}	0.0018(7)	U_{12}	U_{12}
K(4)	12*b*	0.5371(1)	0.7849(1)	0.3654(1)	0.0374(6)	0.0395(6)	0.0343(6)	0.0002(4)	0.0005(4)	0.0059(5)
O(1)	4*a*	0.3345(3)	x	x	0.031(1)	0.031(1)	0.031(1)	–0.001(1)	–0.001(1)	–0.001(1)

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