Crystal structure of potassium niobate, $K_6Nb_{10.80}O_{30}$, *a partially filled tetragonal tungsten bronze-type structure*

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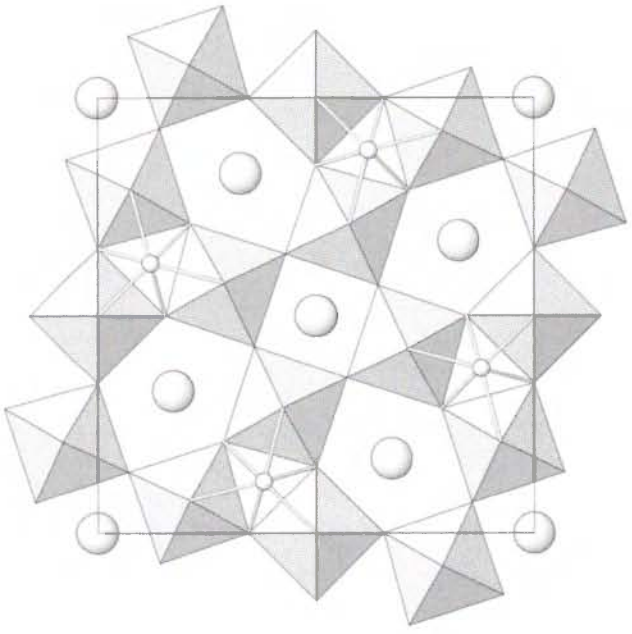

Abstract

$K_6Nb_{10.80}O_{30}$, tetragonal, *P4/mbm* (No. 127), $a = 12.537(2)$ Å, $c = 3.9730(1)$ Å, $V = 624.5$ Å^3, $Z = 1$, $R_{gt}(F) = 0.037$, $wR_{ref}(F^2) = 0.119$, $T = 293$ K.

Source of material

The title compound was prepared in the course of a systematic investigation of the ternary system K_2O–Nb_2O_5–B_2O_3. It crystallizes within a wide range of ternary composition: 20 mol% < x(K_2O) < 35 mol%, 8 mol% < x(B_2O_3) < 50 mol%, 22 mol% < x(Nb_2O_5) < 55 mol%, but does not occur in the binary system K_2O–Nb_2O_5. $K_6Nb_{10.8}O_{30}$ was grown from a melt with molar composition of Nb_2O_5: K_2CO_3 : B_2O_3 of 1 : 0.857 : 1 to colorless, long-prismatic crystals which were separated from the flux using hot diluted hydrochloric acid.

Discussion

The stucture of $K_6Nb_{10.8}O_{30}$ belongs basically to the tetragonal tungsten bronze structure type, which consists of close-packed layers of oxygen atoms with five different possible sites for accommodation of metal cations. Using the nomenclature of Jamieson et al. [1], the complete unit cell of a tetragonal tungsten bronze is $(A1)_2(A2)_4C_4(B1)_2(B2)_8O_{30}$. In $K_6Nb_{10.8}O_{30}$, Nb atoms occupy both of the slightly distorted octahedral sites B1 (Nb1) and B_2 (Nb2) fully. The A1 site with a cube-octahedral 12-fold coordination as well as the A2 site with 9-fold distorted tricapped trigonal prismatic coordination are fully occupied by K atoms. To achieve electrostatic neutrality the tricapped trigonal prismatically coordinated C site is partly by 1/5 occupied by additional niobium Nb3 atoms. The structure is isotypic with $K_6Ta_{10.8}O_{30}$ [2]. Due to the frequent occurrence of ferroic phase transitions in tetragonal tungsten bronze-type structures, thermal analysis investigations (DSC) were performed within the temperature range from 150 K to 870 K. No thermal effect was detected.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.20 × 0.22 × 0.24 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	58.87 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD-4, ω/2θ
$2\theta_{max}$:	70.14°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7310, 818
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 684
$N(param)_{refined}$:	47
Programs:	MolEN [3], SHELXL-97 [4], ATOMS [5]

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Nb(1)	2*c*		0	1/2	1/2	0.0211(4)	U_{11}	0.0227(5)	-0.0109(4)	0	0
Nb(2)	8*j*		0.07572(5)	0.20747(4)	1/2	0.0155(3)	0.0134(3)	0.0317(4)	0.0022(2)	0	0
Nb(3)	4*g*	0.203(5)	0.1203(2)	1/2–*x*	0	0.016(1)	U_{11}	0.010(2)	-0.002(1)	0	0
K(1)	2*a*		0	0	0	0.0294(9)	U_{11}	0.026(1)	0	0	0
K(2)	4*g*		0.3272(1)	1/2–*x*	0	0.0311(7)	U_{11}	0.0184(8)	0.0153(8)	0	0

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Table 2. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	2*d*	0	1/2	0	0.049(5)	U_{11}	0.014(4)	–0.001(7)	0	0
O(2)	8*i*	0.0758(6)	0.2108(5)	0	0.051(4)	0.037(3)	0.008(2)	–0.001(3)	0	0
O(3)	4*g*	0.2119(4)	1/2–x	1/2	0.010(1)	U_{11}	0.049(4)	–0.003(2)	0	0
O(4)	8*j*	0.0000(5)	0.3456(4)	1/2	0.018(2)	0.008(2)	0.095(6)	0.002(2)	0	0
O(5)	8*j*	0.1419(4)	0.0701(3)	1/2	0.015(2)	0.008(2)	0.028(2)	0.002(1)	0	0

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