Crystal structure of tripotassium monosodium ditungstate, $K_3Na[WO_4]_2$

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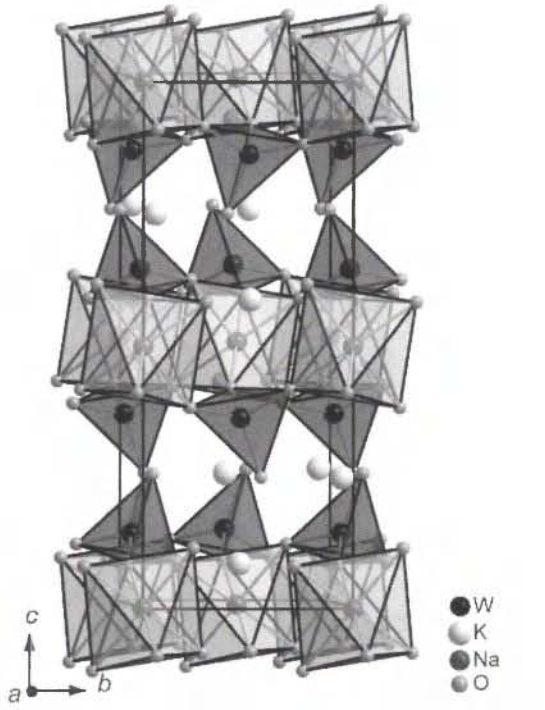

Abstract

$K_3NaO_8W_2$, monoclinic, $C12/c1$ (no. 15), $a = 10.4928(1)$ Å, $b = 6.0693(1)$ Å, $c = 15.2921(1)$ Å, $\beta = 90.087(2)°$, $V = 986.8$ Å^3, $Z = 4$, $R_{gt}(F) = 0.060$, $wR_{ref}(F) = 0.058$, $T = 293$ K.

Source of material

Single crystals of the title compound, $K_3Na(WO_4)_2$, were obtained from the reaction of anhydrous sodium tungstate and anhydrous potassium tungstate. The starting mixture Na_2WO_4 and K_2WO_4 with a molar ratio of 1:3 were mixed intimately. The mixture was pressed into a pellet and subsequently heated at 600 °C for 40 h in a silver crucible.

Experimental details

The unit cell parameters were refined from the obtained X-ray powder diffraction data using Cu $K_{\alpha 1}$ radiation ($\lambda = 1.540543$ Å), and silicon (cubic, $Fd3m$, $a = 5.4308(8)$ Å) was used as an internal standard. The structure refinement succeeded from a merohedral twin by refining six individual domains of the structure.

Discussion

$K_3Na(WO_4)_2$ is reported [1] to crystallize trigonally in space group $P3m1$, isostructural to glaserite, $K_3Na(SO_4)_2$ [2]. From our recent investigations, we found the phase to be isotypic to the room temperature phase of the corresponding molybdates and chromates, $K_3Na(MoO_4)_2$ [3] and $K_3Na(CrO_4)_2$ [4], respectively. The main building units of the crystal structure are WO_4 tetrahedra which are not parallelly aligned to (001) plane and are linked by sodium and potassium cations. Sodium is coordinated by six oxygen atoms in the form of an octahedron while potassium forms polyhedra with 9 and 10 oxygen atoms. At about 275 °C, a phase transition from monoclinic system into trigonal (space group $P3m1$ (no. 164), $a = 6.1305(1)$ Å, $c = 7.6944(1)$ Å, $V = 250.44(7)$ Å^3, $Z = 2$) was observed from high-temperature X-ray measurements. The structure of $K_3Na(WO_4)_2$ above 275 °C is isotypic with the prototypic glaserite, $K_3Na(SO_4)_2$ [2].

Table 1. Data collection and handling.

Crystal:	colorless, irregular, size 0.08 × 0.10 × 0.12 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	246.15 cm^{-1}
Diffractometer, scan mode:	STOE IPDS II, ω/φ
$2\theta_{max}$:	53.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7688, 4027
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\,\sigma(I_{obs})$, 3583
$N(param)_{refined}$:	68
Program:	JANA2000 [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}
W(1)	8*f*	0.1629(1)	0.4795(1)	0.13974(4)	0.0130(3)	0.0156(4)	0.0130(2)	−0.0010(4)	−0.0024(3)	0.0010(4)
K(1)	8*f*	0.1714(5)	0.4759(8)	0.4152(2)	0.024(2)	0.032(3)	0.022(1)	0.001(2)	−0.003(2)	−0.001(2)
K(2)	4*e*	0	0.945(1)	¼	0.032(4)	0.027(3)	0.024(2)	0	−0.001(3)	0
Na(1)	4*a*	0	0	0	0.012(5)	0.027(7)	0.018(3)	0.001(5)	−0.003(5)	0.000(5)
O(1)	8*f*	0.172(2)	0.555(3)	0.2516(7)	0.06(1)	0.04(1)	0.020(5)	−0.003(8)	0.007(9)	−0.002(6)
O(2)	8*f*	0.082(2)	0.693(3)	0.081(1)	0.04(1)	0.03(1)	0.019(8)	0.003(8)	−0.002(8)	0.014(7)
O(3)	8*f*	0.079(2)	0.231(3)	0.121(1)	0.018(9)	0.03(1)	0.04(1)	−0.013(6)	−0.01(1)	0.005(8)
O(4)	8*f*	0.315(1)	0.455(4)	0.093(1)	0.022(8)	0.05(1)	0.03(1)	0.007(8)	0.005(7)	0.01(1)

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