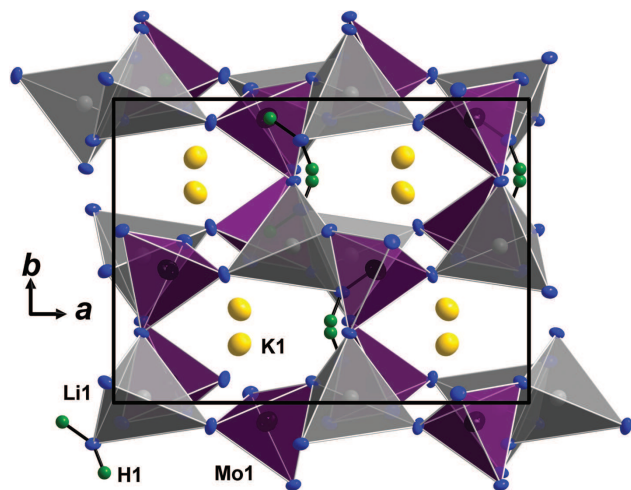


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Redetermination of the crystal structure of potassium lithium molybdate monohydrate, $\text{KLiMoO}_4 \cdot \text{H}_2\text{O}$



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Abstract

$\text{H}_2\text{KLiMoO}_5$, orthorhombic, *Pbca* (no. 61), $a = 10.606(1) \text{ \AA}$, $b = 7.742(1) \text{ \AA}$, $c = 12.845(2) \text{ \AA}$, $V = 1054.8(2) \text{ \AA}^3$, $Z = 8$, $R_{\text{gt}}(F) = 0.0333$, $wR_{\text{ref}}(F^2) = 0.0749$, $T = 298(2) \text{ K}$.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless prism
Wavelength:	Size $0.35 \times 0.11 \times 0.10 \text{ mm}$
μ :	Mo $K\alpha$ radiation (0.71073 \AA)
Diffractometer, scan mode:	32.0 cm^{-1}
$2\theta_{\text{max}}$, completeness:	Bruker APEX-II, φ and ω
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	147.8° , $>95\%$
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	136629 , 10396 , 0.036
$N(\text{param})_{\text{refined}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 8140
Programs:	82
	SHELX [7], Bruker programs [8, 9], SIR92 [10]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Mo1	0.13095(2)	0.06221(2)	0.12578(2)	0.01339(2)
K1	0.19692(3)	−0.19224(3)	0.36849(2)	0.02385(4)
O1	0.01902(9)	−0.06670(11)	0.19304(6)	0.02349(13)
O2	0.04946(10)	0.13568(11)	0.39687(7)	0.02421(13)
O3	0.06711(9)	0.27078(10)	0.10354(7)	0.02185(11)
O4	0.26615(8)	0.07740(13)	0.20569(7)	0.02495(14)
O5	0.17033(10)	−0.03009(12)	0.00666(6)	0.02499(14)
Li1	0.4297(2)	−0.0155(3)	0.17781(16)	0.0194(3)
H1	0.022(3)	0.231(4)	0.3760(16)	0.035(7)*
H2	0.124(4)	0.063(6)	0.377(3)	0.10(2)*

Source of material

K_2CO_3 (6.91 g), Li_2CO_3 (3.69 g) and MoO_3 (14.394 g) were dissolved in distilled water (100 g). This solution was slowly evaporated. Small colorless crystals were obtained after a week.

Experimental details

All H atoms were located in a difference fourier map and refined isotropically without any restraints.

Discussion

In contrast to the literature-known structure of the title compound [1] the two hydrogen atoms of the water molecule have been located in the course of this structure determination.

The MoO₄ and LiO₄ tetrahedra share a corner. These two tetrahedra build a zig-zag chain along the b-axis. Between these chains, potassium cations and water molecules are located. Each tetrahedron is slightly distorted. Compared to the rubidium compound [1], the lattice parameters are shorter, because the radius of potassium is smaller than Rb. Li-O bond distances are more or less similar within the error range.

Contrary to this hydrous compound, the anhydrous compound, KLiMoO₄ crystallizes in hexagonal system [2, 3]. Hydrogen bonding occurs between water molecules and the oxygen atoms of the LiO₄ and MoO₄ tetrahedra. The bond length between the donor and the hydrogen atom is about 0.83 Å and the distance between the acceptor and the hydrogen atom is 2.67 Å. The O-H...O angle is 169(3)°. According to the hydrogen bond classification [4, 5], the hydrogen bond in the title compound lies between the strong and moderate hydrogen bond. Based on the X-ray result, the H-O-H angle in this compound is 133(3)°. Actually, the ideal water molecule should show about 105.9 degree and in the previous report on CsMnCl₃·2H₂O by neutron diffraction [6], this angle is 105.94 degree. This strong deviation from the ideal value of the free water can result from the hydrogen bond in the title compound, because this hydrogen bond can attract the hydrogen atoms belonging to water molecule to the oxygen atom which is shared by LiO₄ and MoO₄ tetrahedra.

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