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Synthesis, characterization and crystal structure of $K_2Bi(PO_4)(MoO_4)$

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Abstract

A new potassium bismuth phosphate-molybdate $K_2Bi(PO_4)(MoO_4)$ has been synthesized by the flux method and characterized by single-crystal and powder X-ray diffraction, IR spectroscopic studies. The compound crystallizes in the orthorhombic system with the space group *Ibca* and the cell parameters: $a = 19.7037(10)$, $b = 12.4752(10)$, $c = 7.0261(10)$. This phase exhibits an original layered structure, in which the $[Bi(PO_4)(MoO_4)]_\infty$ layers consist of $[Bi_2Mo_2O_{18}]_\infty$ chains linked through single PO_4 tetrahedra. The K^+ cations interleaved between these layers exhibit a monocapped distorted cubic coordination.

Keywords: Phosphate-molybdate; Crystal structure; Layered structure; $K_2Bi(PO_4)(MoO_4)$; IR spectra

1. Introduction

Complex oxides containing bismuth have many interesting and useful properties and they are known as oxygen ion superconductors, for example, $Bi_4V_2O_{11}$ [1–3], three-dimensional antiferromagnetic materials—Bi_2CuO_4 [4], selective oxidation catalysts [5], NLO-materials [6]. It is well established that ions such as Bi^{3+} and Pb^{2+} often generate distorted structures due to the electrostatic effect of the lone pair of electrons. The compounds formed by combination of such ions with tetrahedral anions (MoO_4, WO_4, VO_4, PO_4, etc.) are promising materials in the field of inorganic material technology and have possible applications as hosts for laser materials.

In this context, many works have been devoted to mixed bismuth phosphates and molybdates. The study of several pseudo-binary and pseudo-ternary systems as Bi_2O_3–P_2O_5, $M^I_2O - Bi_2O_3 - P_2O_5$, $M^{II}O$–Bi_2O_3–P_2O_5 has allowed to point out and characterize new oxide-phosphates, e.g., $Bi_{6.67}O_4(PO_4)_4$ [7], $M^I_{0.5}Bi_{6.5}O_4(PO_4)_4$ (M^I = Li, Na, K) [8], $Na_3Bi_5(PO_4)_6$ [9], $Ba_3Bi(PO_4)_3$ [6], $M^{II}Bi_6P_4O_{20}$ (M^{II} = Ca, Sr, Ba, Cd, Pb) [8,10], $M^{II}BiPO_5$ (M^{II} = Mn, Co, Ni,) [5,11,12], $M^{II}_2BiPO_6$ (M^{II} = Mg, Ca, Cu, Zn, Cd, Pb, and others) [13,14], $Bi_2PbMnO_4(PO_4)_2$ [15], $BiMn_6PO_{12}$ [16]. These compounds can be described in the terms of association of discrete PO_4 tetrahedra sharing vertices with MO_X polyhedra. Using this model Bi^{3+} plays a main role within the created frameworks. Nevertheless, this classical description does not allow establishing topological relationships between the structures and is not well suited to describe recently evidenced disordered materials.

This paper describes the preparation and structural approach a new bismuth phosphate-molybdate of $K_2Bi(PO_4)(MoO_4)$.

2. Experimental

2.1. Synthesis

The title compound has been prepared by the flux method. All reagents were analytical grade (Merck). A mixture of K_2MoO_4 (5.61 g, 23.58 mmol), MoO_3 (3.39 g, 23.55 mmol), Bi_2O_3 (2.71 g, 5.81 mmol), KPO_3 (1.37 g, 11.61 mmol) and $K_4P_2O_7$ (1.92 g, 5.81 mmol) was heated to 1000 °C in a platinum crucible. The mixture was kept at this temperature until homogenization liquid (40 min) and then cooled to 650 °C at a rate of $50\,^\circ C\,h^{-1}$. The product

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was collected as colorless crystals, the linear dimensions of which typically exceed 0.1 mm.

2.2. Analytical procedures

The ICP determination of the potassium, phosphorus, bismuth and molybdenum in the prepared material was performed on a "Spectroflame Modula ICP" ("Sectro", Germany) instrument. Analysis showed that K/P/Mo/Bi mol ratio was 2:1:1:1. The infrared spectra were recorded with a NICOLET Nexus 470 (FTIR) spectrometer, in the spectral range 400–4000 cm^{-1} (compound dispersed in KBr).

2.3. X-data collection and structure refinement

Initial determination of unit cell parameters and space group were performed by X-ray powder diffraction. The X-ray diffraction pattern was collected using a conventional powder diffractometer Siemens D500 operating in Bragg–Brentano ($\theta/2\theta$) geometry using (Niβ-filtered Cu *K*α radiation). The sample of $K_2Bi(PO_4)(MoO_4)$ was gently ground in agate mortar and the data were collected at room temperature over the angular range 5–85° 2θ with a step of 0.01° and a count time of 10 s/step. The pattern was indexed using the program DICVOL04 [17] from the angular positions of 20 reflections. The compound crystallizes in the orthorhombic system with the space group *Ibca* (no. 73) and the cell parameters: $a = 19.7037(10)$ Å, $b = 12.4752(10)$ Å, $c = 7.0261(10)$ Å (figures of merit: $M_{20} = 19.7$ [18] and $F_{20} = 29.7$ (0.0068, 99) [19]). Fig. 1 shows the observed, calculated and difference X-ray profiles of powder diffraction for this phosphate-molybdate.

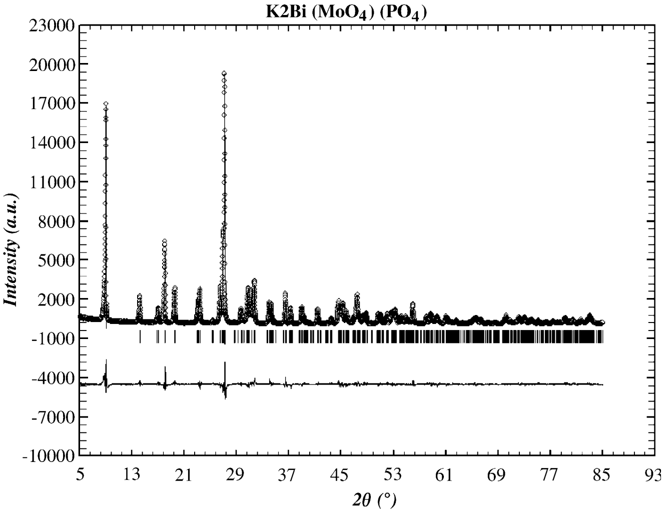

Fig. 1. Powder X-ray diffraction pattern and Rietveld analysis for $K_2Bi(PO_4)(MoO_4)$. Circle signs correspond to observed data; the solid line is the calculated profile. Tick marks represent the positions of allowed reflections, and a difference curve on the same scale is plotted at the bottom of the pattern.

A single crystal of 0.1 × 0.1 × 0.1 mm dimensions was used for indexing and intensity data collection on an Oxford-Diffraction "XCalibur 3" diffractometer with 2048 × 2048 K (4MPixel) CCD detector monochromatized Mo*K*α radiation ($\lambda = 0.7107$ Å). Data were collected using an φ and ω scan method. A total of 14027 reflections were collected with a maximum θ of 28°, of which of 1044 reflections were independent. An experiment was performed at 293 K. Crystallographic data and structure refinement parameters for $K_2Bi(PO_4)(MoO_4)$ are reported in Table 1.

The structure was solved using a heavy atom method and refined by full-matrix least-squares in the anisotropic approximation using the SHELXS-97 [20] and

Table 1
Crystallographic data and structure refinement parameters for $K_2Bi(PO_4)(MoO_4)$

Crystal data	
Formula unit	$K_2Bi(PO_4)(MoO_4)$
Formula weight	542.09
Crystal system	Orthorhombic
Space group	*Ibca* (no. 73)
Cell parameter	
a (Å)	19.7037(10)
b (Å)	12.4752(10)
c (Å)	7.0261(10)
V (Å^3)	1727.07(30)
Z	8
ρ_{cal} (g cm^{-3})	4.17
Intensity measurements	
Crystal dimensions (mm)	0.1 × 0.1 × 0.1
Apparatus	XCalibur-3 CCD
Wavelength (Å)	(Mo *K*α) = 0.71073
Monochromator	Graphite
μ (mm^{-1})	22.961
Scan mode	φ and ω scans
Theta range	3.27–28°
Unique reflections, R_{int}	1044, 0.0787
Retained reflections ($F_o > 4\sigma F_o$)	1000
h_{range}	$-26 \rightarrow 26$
k_{range}	$-16 \rightarrow 16$
l_{range}	$-7 \rightarrow 9$
F (000)	1936
Structure solution and refinement	
Absorption correction	Integration
$T_{\min}$, $T_{\max}$	0.0198, 0.232
Resolution method	Heavy atom method
Agreement factors	$R_1 = 0.035$; w$R = 0.0773$; $S = 1.199 (F_o > 4\sigma F_o)$
Number parameters	62
Extinction parameters	0.00182(11)
$(\Delta\rho)_{\max,\,\min}$ (e/Å^{-3})	0.328, −2.737
Weighting scheme	$w = 1/[\sigma(F_o^2) + (0.0355P)^2 + 31.7364P]$, where $P = (F_o^2 + 2F_c^2)/3$

SHELXL-97 [21] program packages. The final values were $R_1 = 0.035$ and $wR = 0.0773$ for all independent reflections (62 refined parameters). The residual electron density max/min is $-0.328/2.737\,\mathrm{e}/\text{Å}^{-3}$. The coordinates of the atoms are listed in Table 2. The bond lengths and bond angles in the coordination polyhedra for $K_2Bi(PO_4)(MoO_4)$ are listed in Table 3.

Further details of the crystal structure of $K_2Bi(PO_4)(MoO_4)$ investigation are available from the Fachionformationszentrum Karlsruhe, D-76344 Eggeinstein–Leopoldshafen (Germany), on quoting depository number CSD-416427.

3. Results and discussion

The crystallographic study reveals that the title compound possesses a two-dimensional framework structure of vertex-sharing BiO_8 dodecahedra, PO_4 and MoO_4 tetrahedra. This phase can be formulated as a complex potassium bismuth (III) phosphate-molybdate $K_2Bi(PO_4)(MoO_4)$.

The bismuth atom is surrounded by eight oxygen atoms and exhibits distorted dodecahedral coordination, with two shorter Bi–O distances 2.349(6) Å, two pairs of longer

Table 2
The atomic coordinates and their equivalent isotropic thermal parameters for $K_2Bi(PO_4)(MoO_4)$

Atom	Site	*x*	*y*	*z*	U_{eq} (Å^2)
Bi1	8*d*	1/4	0.58076(2)	0	0.01127(18)
K1	16*f*	0.09441(9)	0.83076(14)	0.2163(3)	0.0303(4)
Mo1	8*c*	0.08133(4)	1/2	1/4	0.0185(2)
P1	8*d*	1/4	0.82328(18)	0	0.0105(5)
O1	16*f*	0.2385(3)	0.9005(4)	0.1689(8)	0.0193(10)
O2	16*f*	0.3118(3)	0.7499(4)	0.0277(7)	0.0177(10)
O3	16*f*	0.1319(3)	0.5332(4)	0.0468(9)	0.0268(11)
O4	16*f*	0.0310(3)	0.3901(5)	0.1907(10)	0.0397(15)

Table 3
The bond lengths (Å) and bond angles (°) in the coordination polyhedra for $K_2Bi(PO_4)(MoO_4)$

Bi(1)O_8 polyhedra					
Bi(1)–O(1)	2.349(6) × 2	O(1)^{i}–Bi(1)–O(1)^{ii}	91.17(14)	O(1)^{iv}–Bi(1)–O(3)^{v}	78.84(18)
	2.553(5) × 2	O(1)^{ii}–Bi(1)–O(1)^{iv}	123.82(18)	O(1)^{vi}–Bi(1)–O(2)	97.93(12)
Bi(1)–O(2)	2.444(5) × 2	O(1)^{ii}–Bi(1)–O(2)^{v}	92.35(17)	O(1)^{vi}–Bi(1)–O(2)^{v}	108.54(12)
Bi(1)–O(3)	2.424(6) × 2	O(1)^{ii}–Bi(1)–O(3)	89.00(21)	O(1)^{vi}–Bi(1)–O(3)	80.22(15)
		O(1)^{iii}–Bi(1)–O(1)^{iv}	92.49(13)	O(2)–Bi(1)–O(2)^{v}	60.61(17)
		O(1)^{iii}–Bi(1)–O(2)	60.29(14)	O(2)–Bi(1)–O(3)	132.76(18)
		O(1)^{iii}–Bi(1)–O(2)^{ii}	118.36(14)	O(2)^{v}–Bi(1)–O(3)	75.16(19)
		O(1)^{iii}–Bi(1)–O(3)	166.46(14)	O(3)–Bi(1)–O(3)^{v}	151.66(20)
Mo(1)O_4 tetrahedron					
Mo(1)–O(3)	1.790(6) × 2	O(3)–Mo(1)–O(3)^{vii}	112.33(28)	O(3)–Mo(1)–O(4)^{vii}	109.00(28)
Mo(1)–O(4)	1.743(6) × 2	O(3)–Mo(1)–O(4)	107.95(27)	O(4)–Mo(1)–O(4)^{viii}	110.63(29)
P(1)O_4 tetrahedron					
P(1)–O(1)	1.545(6) × 2	O(1)–P(1)–O(2)	112.99(28)	O(1)–P(1)–O(1)^{v}	110.65(27)
P(1)–O(2)	1.536(6) × 2	O(1)–P(1)–O(2)^{v}	102.86(29)	O(2)–P(1)–O(2)^{v}	106.82(30)
K(1)O_8 polyhedra					
K(1)–O(1)	2.988(6)	O(1)–K(1)–O(2)^{v}	52.49(17)	O(2)^{v}–K(1)–O(3)^{xi}	55.08(13)
K(1)–O(2)^{ii}	2.768(6)	O(1)–K(1)–O(2)^{ii}	62.87(17)	O(2)^{v}–K(1)–O(4)^{vi}	50.23(13)
K(1)–O(2)^{v}	2.715(6)	O(1)–K(1)–O(3)^{viii}	59.27(15)	O(2)^{v}–K(1)–O(4)^{vii}	94.43(17)
K(1)–O(2)^{iv}	2.970(6)	O(1)–K(1)–O(4)^{vii}	101.75(16)	O(2)^{v}–K(1)–O(4)^{viii}	77.76(16)
	3.216(6)	O(1)–K(1)–O(4)^{viii}	101.75(16)	O(3)^{viii}–K(1)–O(3)^{xi}	76.86(11)
	3.095(6)	O(2)^{ii}–K(1)–O(3)^{viii}	120.37(16)	O(3)^{viii}–K(1)–O(4)^{vii}	155.95(16)
	3.207(7)	O(2)^{ii}–K(1)–O(3)^{xi}	45.23(12)	O(3)^{viii}–K(1)–O(4)^{viii}	52.82(16)
	2.661(7)	O(2)^{ii}–K(1)–O(4)^{vii}	78.97(16)	O(3)^{viii}–K(1)–O(4)^{xii}	76.04(11)
		O(2)^{ii}–K(1)–O(4)^{viii}	157.41(17)	O(3)^{xi}–K(1)–O(4)^{vii}	116.69(12)
		O(2)^{ii}–K(1)–O(4)^{xii}	47.51(13)	O(3)^{xi}–K(1)–O(4)^{i}	117.84(12)
		O(2)^{v}–K(1)–O(2)^{ii}	79.68(17)	O(4)^{vii}–K(1)–O(4)^{viii}	103.68(18)
		O(2)^{v}–K(1)–O(3)^{viii}	76.85(16)	O(4)^{vii}–K(1)–O(4)^{xii}	115.67(12)

Symmetry transformations used to generate equivalent atoms: i: x, $1.5-y$, $-0.5+z$; ii: $0.5-x$, $1.5-y$, $0.5-z$; iii: $0.5-x$, $-0.5+y$, z; iv: x, $-0.5+y$, $-z$; v: $0.5-x$, y, $-z$; vi: $0.5-x$, $-0.5+y$, $1+z$; vii: x, $1-y$, $0.5-z$; viii: x, $0.5+y$, $-z$; ix: x, $1.5-y$, $0.5+z$; x: $-x$, $0.5+y$, $0.5-z$; xi: $1+x$, $1.5-y$, $0.5+z$; xii: $1-x$, $0.5+y$, $0.5-z$.

distances 2.424(6) and 2.444(5) Å, two the longest 2.553(5) Å (Fig. 2(a)). The PO_4 and MoO_4 tetrahedra are essentially regular and have C_{2v} symmetry. The interatomic distances and angles are actually typical, with characteristic P–O and Mo–O separations: two distances $P–O = 1.536(6)$ and 1.545(6) Å, O–P–O angles are spread in the range 102.8(3)–113.0(3)°; two distances $Mo–O = 1.790(6)$ Å and two distances $Mo–O = 1.743(6)$ Å; O–Mo–O angles are spread in the range 107.9(3)–112.4(3)°. The coordination polyhedron of the K^+ cation is monocapped distorted cube formed by eight O atoms assuming a cut-off distance of 3.25 Å (Fig. 2(b)). This polyhedron is very irregular; in fact, the K–O distances range from 2.661(7) to 3.216(6) Å (Table 3).

BiO_8 edge-sharing polyhedra form an infinite parallel zig-zag chain extending in the *c* direction, with a $Bi \cdots Bi$ distance of 4.05 Å. The connection between neighboring BiO_8 dodecahedra of the chain is achieved by intermediate MoO_4 tetrahedra: Bi–O–Mo bond angle is 134.4(3)°. The similar principle for connection of BiO_8 dodecahedra and $Mo(2)O_4$ tetrahedra was observed for $RbBi(MoO_4)_2$ [23].

Each PO_4 tetrahedra binds the four BiO_8 polyhedra and each bismuth polyhedra in its turn contacts with four phosphate tetrahedra. This tetrahedron forms common edges with two bismuth polyhedra and provide monodentate type of coordination to other two bismuth atoms forming two four-membered planar rings BiO_2PO_2Bi. The values of angles for Bi–O(1)–P, Bi–O(2)–P and Bi–P–Bi are 100.3(3), 96.3(3) and 180.0(1)°, respectively. Geometry of the BiO_8/PO_4 structural fragment is remarkably similar to that of the bismuth phosphates, for example, orthophosphate $BiPO_4$ [22]. The value of the Bi–O–P angle in the case of the phosphate exhibiting monodentate type of coordination is 143.9(3)°.

The structure consists of anionic $[Bi(PO_4)(MoO_4)]^{2-}$ layers parallel to direction *c* interconnected by K^+ cation. The distance between two successive layers is a half of cell dimension *a* (9.852 Å). In the anionic $[Bi(PO_4)(MoO_4)]^{2-}$ layers two different structural units can be distinguished: single PO_4 tetrahedron and unit $Bi_2Mo_2O_{18}$. Unit $Bi_2Mo_2O_{18}$ is build up with two corner-sharing dodecahedra BiO_8 and MoO_4 tetrahedra (Fig. 3(a)). In this representation it is observed the formation of $[Bi_2Mo_2O_{18}]_\infty$ chains running along *c*. The anionic layers are building up from $[Bi_2Mo_2O_{18}]_\infty$ chains linked through single PO_4 tetrahedra in the *bc*-plane (Fig. 3b). The bismuth and phosphorus atoms are in the 8*d* special positions in this plane; the molybdenum atoms occupy positions 8*c*. The potassium cations occupy positions 16*f* and exist in tubes running along the *c* direction (Fig. 4). The nearest contacts between potassium atoms are 4.05(3) and 4.23(2) Å.

$K_2Bi(PO_4)(MoO_4)$ is isostructural to the orthorhombic modification $Na_2Y(PO_4)(MoO_4)$ [24,25]. Both compounds are similar when it comes to bond distances and distortion of the $M^{III}O_8$ dodecahedra: Y–O distances ranging from 2.259 to 2.428 Å [25] and Bi–O bonds distances of 2.349–2.553 Å (Table 3). In contrast to the latter those compounds exhibit different coordination environment of alkaline metals. The sodium atom is surrounded by six oxygen atoms (Na–O distances ranging from 2.283 to 2.553 Å) and the potassium atom is eight-coordinated (Table 3). Very similar alternating anionic layers and arrangement of calcium cation for natural compound $Ca_2Y(AsO_4)(WO_4)_2$ is observed [26]. At the same time the structure of two-dimensional anionic frameworks ($[Y(AsO_4)(WO_4)_2]^{4-}$ and $[Bi(PO_4)(MoO_4)]^{2-}$) are different.

The IR spectrum of the title compound is shown in Fig. 5. The characteristic bands confirm the simultaneous presence of the phosphate and molybdate groups. The local symmetry of PO_4^{3-} and MoO_4^{2-} tetrahedra are C_{2v} and exhibit all the bands predicted by group theory. The space group of this compound is *Ibca* (no. 73). By assuming separation of the vibration into internal and external modes, factor group analysis led to the following internal modes of the PO_4 and MoO_4 anions. Thus, we expect v_1–*F*

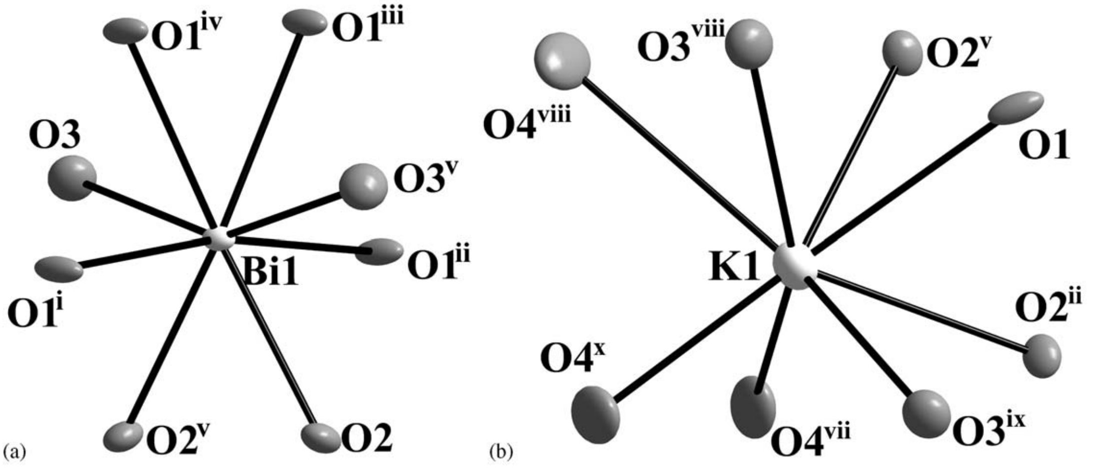

Fig. 2. The oxygen atom coordination around (a) the bismuth and (b) the potassium atoms (symmetry codes: (i) x, $1.5-y$, $-0.5+z$; (ii) $0.5-x$, $1.5-y$, $0.5-z$; (iii) $0.5-x$, $-0.5+y$, z; (iv) x, $-0.5+y$, $-z$; (v) $0.5-x$, y, $-z$; (vii) x, $1-y$, $0.5-z$; (viii) x, $0.5+y$, $-z$; (ix) x, $1.5-y$, $0.5+z$; (x) $-x$, $0.5+y$, $0.5-z$).

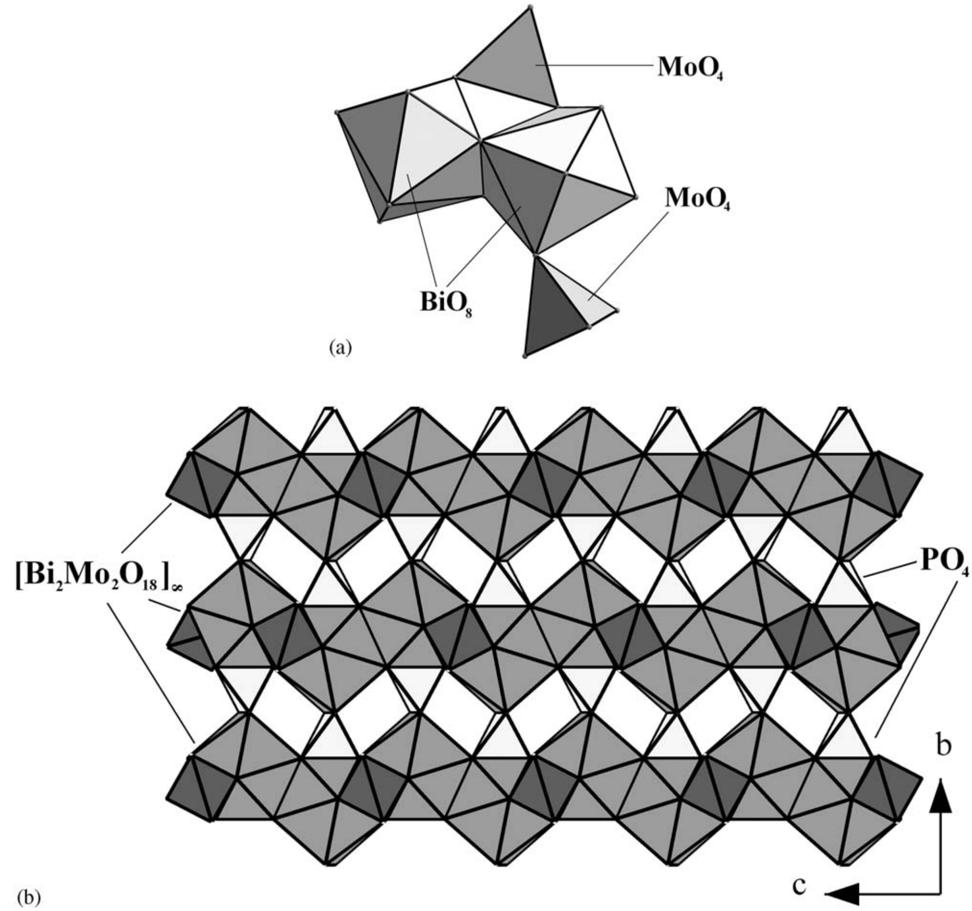

Fig. 3. (a) The unit $Bi_2Mo_2O_{18}$ and (b) the $[Bi_2Mo_2O_{18}]_\infty$ chains linked through single PO_4 tetrahedra.

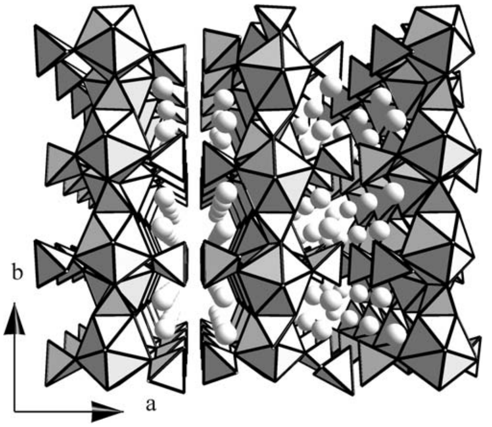

Fig. 4. Projection of the structure of $K_2Bi(PO_4)(MoO_4)$ along *c*.

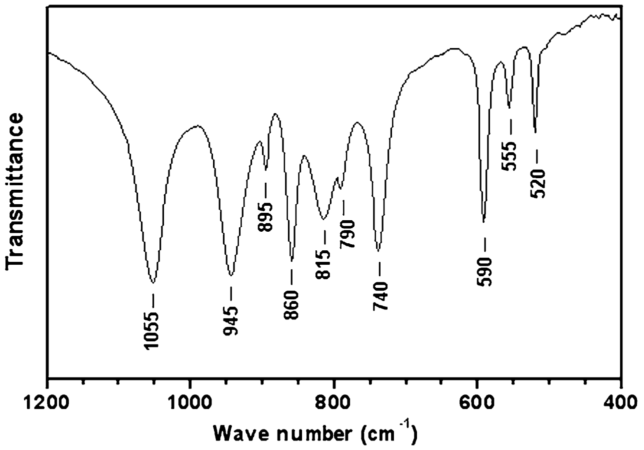

Fig. 5. IR spectrum of $K_2Bi(PO_4)(MoO_4)$.

and ν_3–3*F* for the IR active stretching vibrations, and ν_2–2*F* and ν_4–3*F* for the bending vibrations.

The single intensive band at 1055 cm^{-1}, may be ascribed to the asymmetric stretching vibration $\nu_3(F_2)$ in PO_4^{3-} tetrahedron. The remaining band at 945 cm^{-1} can be most likely assigned to the symmetric $\nu_1(A_1)$ PO_4 assuming the asymmetric $\nu_3(F_2)$ MoO_4 tetrahedra. The bands in the range 900–740 cm^{-1} are due to the Mo–O stretching frequencies in the MoO_4 tetrahedron: $\nu_3(F_2)$–815 and 860 cm^{-1}, $\nu_1(A_1)$–weak 895 cm^{-1}. The 520–600 cm^{-1} region shows three bonds expected for $\nu_4(F_2)$ of PO_4 tetrahedron bending vibrations: 590, 555 and 522 cm^{-1}.

4. Conclusions

A potassium bismuth phosphate-molybdate $K_2Bi(PO_4)(MoO_4)$ with an original layered structure has been synthesized and characterized by powder X-ray diffraction, single-crystal X-ray diffraction and IR spectroscopic studies. Although the number of phosphate-molybdates that have been synthesized up to now is very small, this study suggests that there should exist family of mixed-anionic compounds with general formula $M_2\mathrm{Ln}(\mathrm{PO}_4)(\mathrm{MoO}_4)$ ($M = \mathrm{Na}$, K; $Ln =$ rare earth element).

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