JOURNAL OF SOLID STATE CHEMISTRY 58, 157–163 (1985)

Crystal Structures of Phosphomolybdyl Salts: $Pb(MoO_2)_2(PO_4)_2$ and $Ba(MoO_2)_2(PO_4)_2$

R. MASSE, M. T. AVERBUCH-POUCHOT, AND A. DURIF

Laboratoire de Cristallographie, Centre National de la Recherche Scientifique, Laboratoire associé à l'USMG, 166 X, 38042 Grenoble Cédex, France

Received July 11, 1984; in revised form November 12, 1984

Crystal structures of $Pb(MoO_2)_2(PO_4)_2$ and $Ba(MoO_2)_2(PO_4)_2$ were determined. Both compounds contain the molybdyl group MoO_2. The monoclinic unit-cell parameters are $a = 6.353(7)$, $b = 12.289(4)$, $c = 11.800$ Å, $\beta = 92°56(6)$, and $Z = 4$ for the lead salt and $a = 6.383(8)$, $b = 7.142(7)$, $c = 9.953(8)$ Å, $\beta = 95°46(8)$, and $Z = 2$ for the barium salt. $P2_1/c$ is the common space group. The R values are respectively $R = 0.027$ and $R = 0.031$ for 1964 and 1714 independent reflections. The frameworks built up by a three-dimensional network of monophosphate PO_4 and molybdyl MoO_2 groups are similar, characterized mainly by corner-sharing PO_4 and MoO_6 polyhedra. Two oxygen atoms of each MoO_6 group are bonded to the molybdenum atom only as in other molybdyl salts.

Introduction

Some phosphates containing MoO_2 or WO_2 radicals have been prepared and their structures solved by Kierkegaard (*1*). The two oxygen atoms of MoO_2 and WO_2 radicals are not shared with the associated cations. Therefore we have two very short metal–oxygen distances, meaning that coexistence of ionocovalent and covalent bonds is possible in the same crystal.

Chemical Preparation

The $Pb(PO_3)_2$–MoO_3 phase diagram indicates a compound with a 1 : 1 chemical composition (*3*). All attempts to prepare this phase have produced a compound with a 1 : 2 composition corresponding to the formula $Pb(MoO_2)_2(PO_4)_2$, whatever the MoO_3 composition range may be. This phase diagram is probably erroneous. The reaction

$$\tfrac{1}{2}Pb_2P_4O_{12} \cdot 4H_2O + 2MoO_3 \xrightarrow{600°C} Pb(MoO_2)_2(PO_4)_2 + 4H_2O$$

leads to the definite compound. Analogously,

$$Ba(PO_3)_2 + 2MoO_3 \xrightarrow{600°C} Ba(MoO_2)_2(PO_4)_2,$$

the barium salt, which is easily obtained. Crystals of $Pb(MoO_2)_2(PO_4)_2$ are obtained in a melt of $Pb(PO_3)_2$ kept at 650°C for a day. Crystals of $Ba(MoO_2)_2(PO_4)_2$ are obtained from a melt of $Ba(PO_3)_2$ kept at 650°C for 3 days. The crystals are yellow plates in both cases.

Crystal Data

For these compounds approximate unit-cell parameters and space groups have been determined by single crystal film tech-

0022-4596/85 $3.00

TABLE I

X-RAY POWDER DIAGRAM OF $Pb(MoO_2)_2(PO_4)_2$

$h\,k\,l$	d_{obs}	d_{calc}	I_{obs}
1 0 0	6.36	6.35	13
0 2 0	6.15	6.15	3
0 0 2	5.89	5.89	2
1 1 0	5.63	5.64	2
0 1 2	5.31	5.31	2
$\bar{1}$ 1 1	5.16	5.17	2
1 1 1	5.01	5.01	2
0 2 2	4.25	4.25	3
1 0 2	4.23	4.23	19
$\bar{1}$ 2 1	4.18	4.18	21
1 2 1	4.09	4.09	3
0 2 3	3.31	3.31	100
2 1 0	3.07	3.07	44
2 0 1	3.03	3.03	8
0 4 1	2.980	2.978	11
1 2 3	2.875	2.893	3
0 1 4	2.866	2.865	3
$\bar{2}$ 0 2	2.847	2.846	4
$\bar{2}$ 2 1	2.766	2.767	16
$\bar{1}$ 0 4	2.722	2.717	21
$\bar{1}$ 1 4	2.652	2.654	2
1 0 4	2.630	2.629	4
$\bar{1}$ 2 4	2.486	2.486	14
0 1 5	2.312	2.315	7

niques. The only extinctions, $h0l$ with $l = 2n$ and $0k0$ with $k = 2n$, lead unambiguously to the $P2_1/c$ space group. The angular data obtained from low scan speed powder diffractograms (Tables I and II) were used to refine the cell parameters:

$Pb(MoO_2)_2(PO_4)_2$: $a = 6.353(6)$, $b = 12.31(1)$, $c = 11.795(8)$ Å; $\beta = 92.48(4)$, $Z = 4$, $dx = 4.64$

$Ba(MoO_2)_2(PO_4)_2$: $a = 6.394(2)$, $b = 7.152(2)$, $c = 9.962(4)$ Å; $\beta = 95°42(2)$, $Z = 2$, $dx = 4.29$

The unit-cell dimensions reported above differ slightly from those obtained from the four-circle diffractometer data collection mentioned in the abstract and used throughout the crystal structure determination and the final distance calculations.

Crystal Structure Determinations

Technical parameters concerning the X-ray diffraction data collections are reported in Table III. Lorentz and polarization corrections were applied. Spherical absorption correction was applied for $Ba(MoO_2)_2(PO_4)_2$ ($\mu R = 1.75$). In both cases a unitary weighting scheme has been used throughout the process of crystal structure determination.

TABLE II

X-RAY POWDER DIAGRAM OF $Ba(MoO_2)_2(PO_4)_2$

$h\,k\,l$	d_{obs}	d_{calc}	I_{obs}
0 0 1	6.36	6.36	16
1 1 0	5.80	5.80	11
2 0 0	4.96	4.96	5
0 1 1	4.76	4.75	8
1 1 1	4.17	4.17	74
$\bar{2}$ 0 1	4.10	4.10	70
2 0 1	3.74	3.74	2
0 2 0	3.57	3.57	100
1 2 0	3.36	3.36	4
2 1 1	3.32	3.32	2
0 0 2	3.18	3.18	25
0 2 1	3.12	3.12	6
$\bar{3}$ 1 0	3.00	3.00	97
$\bar{1}$ 1 2	2.857	2.858	26
$\bar{3}$ 1 1	2.809	2.809	23
1 1 2	2.727	2.727	15
$\bar{2}$ 2 1	2.694	2.696	50
3 1 1	2.629	2.629	7
2 2 1	2.586	2.586	4
2 0 2	2.571	2.570	24
4 1 0	2.343	2.343	14
1 3 0	2.317	2.318	7
4 0 1	2.240	2.240	34
$\bar{3}$ 2 1	2.217	2.217	5
$\bar{2}$ 2 2	2.206	2.205	13
1 3 1	2.162	2.163	17
2 3 0	2.148	2.149	5
0 0 3	2.122	2.122	5
$\bar{2}$ 3 1	2.063	2.061	9
$\bar{4}$ 0 2	2.052	2.052	7
$\bar{1}$ 1 3	2.029	2.029	37

TABLE III

EXPERIMENTAL CONDITIONS USED DURING THE X-RAY DATA COLLECTIONS FOR $Pb(MoO_2)_2(PO_4)_2$ AND $Ba(MoO_2)_2(PO_4)_2$

	$Pb(MoO_2)_2(PO_4)_2$	$Ba(MoO_2)_2(PO_4)_2$
Apparatus	Enraf–Nonius CAD4	Enraf–Nonius CAD4
Monochromator	Graphite plate	Graphite plate
Wavelength	$AgK_{\bar{\alpha}}$(0.56083 Å)	$AgK_{\bar{\alpha}}$(0.56083 Å)
Scan mode	ω	ω
Scan speed (°/sec)	From 0.009 to 0.04	From 0.012 to 0.028
Total background measurement (sec)	From 16 to 72	From 21 to 50
Scan width (°)	1°20′	1°20′
θ range (°)	3–25	3–28
Intensity reference reflections	$2\bar{4}4$, $2\bar{4}\bar{4}$	046, 046
Number of collected reflections	1983	2344
Crystal size (mm)	0.13 × 0.13 × 0.13	0.31 × 0.31 × 0.31
μ (cm^{-1})	257	116.8

TABLE IV

FINAL ATOMIC COORDINATES AND EQUIVALENT ISOTROPIC THERMAL PARAMETERS, WITH esds IN PARENTHESES FOR $Pb(MoO_2)_2(PO_4)_2$ $\left(B_{eq} = \frac{4}{3}\sum_i\sum_j a_i a_j \beta_{ij}\right)$

	X	Y	Z	B_{eq} (Å^2)
Pb	0.02431(5)	0.37740(3)	0.23488(2)	1.297(6)
Mo1	0.25540(8)	0.12866(5)	0.09581(4)	0.43(1)
Mo2	0.74089(8)	0.11607(5)	0.40192(4)	0.44(1)
O1	0.2488(9)	0.0093(5)	0.1697(5)	1.01(12)
O2	0.7769(8)	0.3660(5)	0.0446(4)	1.04(12)
O3	0.2890(9)	0.2243(5)	0.2003(5)	1.03(13)
O4	0.7426(9)	0.2450(5)	0.3486(5)	1.19(13)
P1	0.2414(2)	0.1239(2)	0.4570(1)	0.40(3)
P2	0.7581(3)	0.0675(1)	0.1099(1)	0.45(4)
O11	0.0512(8)	0.0867(4)	0.3777(4)	0.68(11)
O12	0.2364(8)	0.4350(4)	0.0715(4)	0.66(11)
O13	0.2342(8)	0.2467(4)	0.4717(4)	0.76(11)
O14	0.4392(8)	0.0880(5)	0.3952(5)	0.77(11)
O21	0.7392(8)	0.0429(5)	0.2369(4)	0.75(11)
O22	0.9386(8)	0.1485(4)	0.0929(5)	0.76(11)
O23	0.1924(8)	0.4567(4)	0.4407(5)	0.87(11)
O24	0.5545(7)	0.1189(5)	0.0579(4)	0.74(10)

(*1*) *$Pb(MoO_2)_2(PO_4)_2$*

A three-dimension Patterson function revealed the atomic positions of Pb, Mo_1, and Mo_2. Fourier synthesis showed the locations of phosphorus and oxygen atoms. The

TABLE V

ANISOTROPIC THERMAL PARAMETERS CORRESPONDING TO THE FACTOR $T = \beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl$ AND STANDARD DEVIATIONS FOR $Pb(MoO_2)_2(PO_4)_2$

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Pb	0.00946(5)	0.00145(5)	0.00273(1)	−0.00186(7)	0.00346(4)	−0.00092(4)
Mo1	0.00296(8)	0.00054(2)	0.00087(2)	0.00003(10)	−0.00002(8)	0.0000(6)
Mo2	0.00258(8)	0.00065(3)	0.00095(2)	−0.00003(10)	0.00020(8)	−0.00004(6)
O1	0.0071(11)	0.0014(3)	0.0018(3)	−0.0006(10)	−0.0016(11)	0.0009(6)
O2	0.0075(10)	0.0017(3)	0.0015(3)	0.00003(11)	−0.0004(9)	0.0012(6)
O3	0.0066(11)	0.0017(3)	0.0017(3)	0.0006(10)	−0.0013(10)	−0.0008(6)
O4	0.0063(11)	0.0012(3)	0.0033(4)	−0.0000(10)	0.0014(11)	0.0012(6)
P1	0.00251(26)	0.00050(7)	0.00088(8)	−0.00002(34)	−0.00004(25)	−0.00010(19)
P2	0.00276(30)	0.00074(9)	0.00083(9)	−0.00016(30)	−0.00017(30)	−0.00006(16)
O11	0.0014(9)	0.0017(3)	0.0013(3)	−0.0009(9)	−0.0011(9)	−0.0010(5)
O12	0.0045(9)	0.0010(3)	0.0011(3)	−0.0010(9)	0.0000(9)	−0.0004(5)
O13	0.0063(10)	0.0004(2)	0.0018(3)	−0.0007(9)	0.0008(9)	−0.0006(5)
O14	0.0023(9)	0.0018(3)	0.0015(3)	−0.0004(9)	0.0010(9)	−0.0003(5)
O21	0.0052(10)	0.0015(3)	0.0009(3)	0.0002(9)	−0.0005(9)	−0.0004(5)
O22	0.0038(9)	0.0007(3)	0.0024(3)	−0.0003(8)	−0.0004(9)	0.0002(5)
O23	0.0063(10)	0.0010(3)	0.0017(3)	−0.0009(9)	−0.0005(10)	0.0010(5)
O24	0.0028(8)	0.0012(3)	0.0019(3)	−0.0002(10)	−0.0001(8)	0.0002(6)

TABLE VI

POSITIONAL PARAMETERS AND THEIR ESTIMATED STANDARD DEVIATIONS FOR $Ba(MoO_2)_2(PO_4)_2$

	X	Y	Z	B_{eq} (Å²)
Ba	0.0	0.0	0.0	0.791(8)
Mo	0.27632(6)	0.46559(5)	0.17490(4)	0.419(6)
P	0.2340(2)	0.0515(2)	0.3426(1)	0.38(2)
O1	0.0413(5)	0.9240(5)	0.3124(4)	0.76(7)
O2	0.2143(6)	0.3374(5)	0.9721(3)	0.66(6)
O3	0.2528(6)	0.1747(5)	0.2181(3)	0.77(7)
O4	0.5700(5)	0.4226(5)	0.1418(3)	0.61(6)
O5	0.6757(6)	0.0177(6)	0.1588(4)	0.95(7)
O6	0.7624(6)	0.1813(5)	0.3961(4)	0.92(7)

Note. $B_{eq} = \frac{4}{3} \sum_i \sum_j a_i a_j \beta_{ij}$.

model obtained was refined using the program system SDP (*4*). The *R* value is 0.027 for 1964 independent reflections satisfying the condition $F_o^2 > 4\sigma(F_o^2)$, σF_o^2 = counting statistics. Nineteen reflections which satisfy $F_o - |F_c| > 98$ in a scale ranging from 0 to 4000 are also omitted. The final atomic coordinates are reported in Table IV and anisotropic thermal factors are given in Table V.[1]

(2) $Ba(MoO_2)_2(PO_4)_2$

This structure has been solved using the same strategy. The final *R* value for the set of 1714 reflections satisfying the condition $F_o^2 > 4\sigma(F_o^2)$, $\sigma(F_o^2)$ = counting statistics, is 0.031. Twenty-nine reflections which satisfy $|F_o - |F_c| > 70$ in a scale ranging from 0 to 1800 are omitted. Final atomic coordinates and anisotropic thermal factors are reported in Tables VI and VII.[1]

Structure Descriptions

(1) $Pb(MoO_2)_2(PO_4)_2$

The three-dimensional framework of Pb $(MoO_2)_2(PO_4)_2$ monophosphate is built up of

[1] A table of structure factors is available on request from the authors.

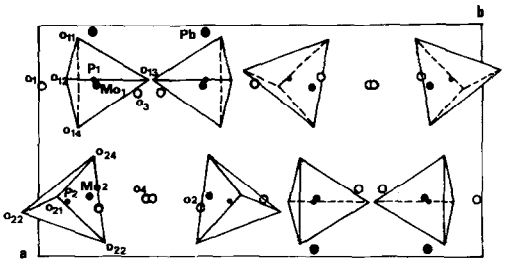

FIG. 1. Simplified (*a*, *b*) projection of $Pb(MoO_2)_2(PO_4)_2$.

PO_4 tetrahedra and MoO_6 octahedra. Each MoO_6 octahedra has two oxygen atoms not shared with other cations (Mo–O = 1.71 Å) and four others (Mo–O = 2.1 Å) shared with four independent PO_4 tetrahedra (Fig. 1). In this way, a PO_4 tetrahedron is in contact with four MoO_6 groups. The structure may be described as chains formed by PO_4 and MoO_6 groups which run along the *c* axis (Fig. 2). The chains with dotted lines

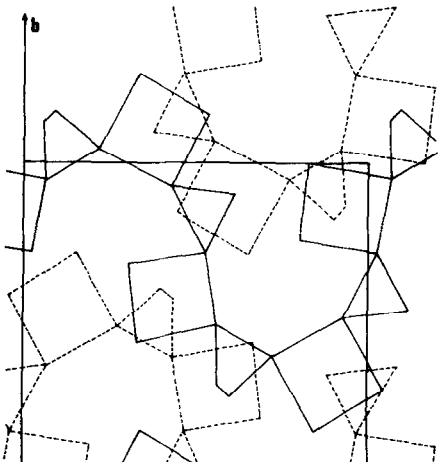

FIG. 2. A (*b*, *c*) projection of $Pb(MoO_2)_2(PO_4)_2$. Only chains of oxygen atoms are presented. This schematic drawing shows chains at $x = 0.25$ (dotted lines) and $x = 0.75$ (full lines).

TABLE VII

ANISOTROPIC THERMAL PARAMETERS CORRESPONDING TO THE FACTOR $T = \beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl$ AND STANDARD DEVIATIONS FOR $Ba(MoO_2)_2(PO_4)_2$

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Ba	0.00596(7)	0.00376(5)	0.00172(3)	−0.0021(1)	0.00197(7)	0.00054(7)
Mo	0.00296(5)	0.00190(4)	0.00109(2)	−0.00025(8)	0.00172(6)	−0.00008(5)
P	0.0023(2)	0.0019(1)	0.00102(7)	0.0005(3)	0.0014(2)	0.0001(2)
O1	0.0026(6)	0.0042(5)	0.0027(3)	−0.0007(9)	0.0024(6)	−0.0019(6)
O2	0.0056(6)	0.0035(5)	0.0011(2)	−0.0008(9)	0.0018(6)	0.0004(6)
O3	0.0086(7)	0.0024(5)	0.0012(2)	0.0011(10)	0.0020(7)	0.0006(6)
O4	0.0025(5)	0.0032(5)	0.0021(2)	−0.0004(9)	0.0024(6)	−0.0002(6)
O5	0.0064(6)	0.0068(6)	0.0012(2)	0.0034(11)	0.0015(7)	0.0009(6)
O6	0.0057(7)	0.0032(5)	0.0031(3)	−0.0007(10)	0.0023(8)	−0.0010(7)

are situated at $x = 0.25$. The chains with full lines are at $x = 0.75$. The chains show a schematic arrangement of MoO_6 octahedra with four oxygen atoms and one molybdenum atom at $x = 0.25$ or $x = 0.75$ and PO_4 tetrahedra with two oxygen and phosphorus atoms at $x = 0.25$ or $x = 0.75$. The phosphorus and molybdenum atoms are omitted from the schematic drawing (Fig. 2). The other two oxygen atoms of PO_4 tetrahedra are shared with MoO_6 octahedra to form chains which run along the a axis (Fig. 3). We can see easily in Fig. 2 the tunnels formed by this framework. The tunnels are occupied at $x \sim 0$ by the Pb atoms. Eight oxygen atoms make up a very deformed antiprism around the Pb atom. This structure is similar in many aspects to the monophosphate $NaMoO_2PO_4$ (*2*) described by Kierkegaard. We must compare the cell of $Pb(MoO_2)_2(PO_4)_2$ with that of $NaMoO_2PO_4$ ($P2_1/n$, $a = 12.08(1)$, $b = 11.96(1)$, $c = 6.359(5)$ Å, $\beta = 91°19(3)$, $Z = 8$). The (a, c) projection of $NaMoO_2PO_4$ is similar to the (b, a) projection of $Pb(MoO_2)_2(PO_4)_2$. Figure 1 is a simplified (a, b) projection of PO_4 tetrahedra which shows their distribution among the Pb, Mo_1, and Mo_2 cations.

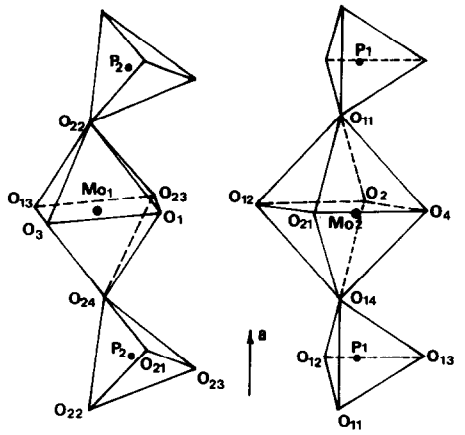

FIG. 3. Parts of chains running in the a direction.

TABLE VIII

INTERATOMIC DISTANCES (Å) AND ANGLES (°) IN THE TWO PHOSPHATE GROUPS FOR $Pb(MoO_2)_2(PO_4)_2$

P1	O11	O12	O13	O14
O11	1.565(3)	110°1(2)	109°4(2)	104°9(2)
O12	2.542(4)	1.537(3)	111°5(2)	109°3(2)
O13	2.519(4)	2.528(4)	1.522(3)	111°5(2)
O14	2.467(4)	2.514(4)	2.536(4)	1.547(3)
P2	**O21**	**O22**	**O23**	**O24**
O21	1.540(3)	110°2(2)	103°5(2)	111°6(2)
O22	2.526(4)	1.540(3)	111°0(2)	107°4(2)
O23	2.409(4)	2.529(4)	1.527(3)	113°2(2)
O24	2.550(4)	2.486(4)	2.564(4)	1.544(3)

TABLE IX

INTERATOMIC DISTANCES (Å) AND ANGLES (°) IN MoO_6 OCTAHEDRA FOR $Pb(MoO_2)_2(PO_4)_2$

Mo1–O1 = 1.710(3)	O1	Mo1	O3	103.1(2)
Mo1–O3 = 1.713(3)	O1	Mo1	O13	166.2(2)
Mo1–O13 = 2.123(3)	O1	Mo1	O22	93.7(2)
Mo1–O22 = 2.029(3)	O1	Mo1	O23	90.4(2)
Mo1–O23 = 2.136(3)	O1	Mo1	O24	96.2(2)
Mo1–O24 = 1.977(3)	O3	Mo1	O13	90.2(2)
	O3	Mo1	O22	91.2(2)
	O3	Mo1	O23	165.9(2)
	O3	Mo1	O24	96.5(2)
	O13	Mo1	O22	82.4(1)
	O13	Mo1	O23	76.0(1)
	O13	Mo1	O24	85.4(1)
	O22	Mo1	O23	84.0(1)
	O22	Mo1	O24	165.6(1)
	O23	Mo1	O24	85.5(1)
Mo2–O2 = 1.707(3)	O2	Mo2	O4	103.9(2)
Mo2–O4 = 1.707(3)	O2	Mo2	O11	94.1(2)
Mo2–O11 = 2.039(3)	O2	Mo2	O12	89.1(2)
Mo2–O12 = 2.254(3)	O2	Mo2	O14	98.7(2)
Mo2–O14 = 1.948(3)	O2	Mo2	O21	161.2(2)
Mo2–O21 = 2.148(3)	O4	Mo2	O11	95.2(2)
	O4	Mo2	O12	165.6(2)
	O4	Mo2	O14	99.9(2)
	O4	Mo2	O21	93.1(2)
	O11	Mo2	O12	77.7(1)
	O11	Mo2	O14	157.0(1)
	O11	Mo2	O21	76.3(1)
	O12	Mo2	O14	83.5(1)
	O12	Mo2	O21	73.1(1)
	O14	Mo2	O21	85.6(1)

Interatomic distances (Å) in PbO_8 polyhedron

Pb–O1 = 2.664(3)	Pb–O11 = 2.930(3)
Pb–O2 = 2.690(3)	Pb–O12 = 2.506(3)
Pb–O3 = 2.572(3)	Pb–O21 = 2.546(3)
Pb–O4 = 2.808(3)	Pb–O23 = 2.790(3)

Distances and angles are summarized in Tables VIII and IX.

(2) $Ba(MoO_2)_2(PO_4)_2$

Here we have the same three-dimensional framework previously described for $Pb(MoO_2)_2(PO_4)_2$. The MoO_6 octahedra shares four oxygen with four PO_4 octahedra (Fig. 4). The two independent oxygen atoms of MoO_6 have distances Mo–O = 1.703 and 1.695 Å. MoO_6 and PO_4 groups form

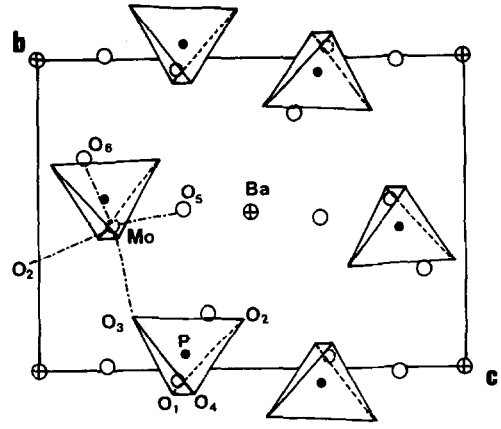

FIG. 4. A (*b*, *c*) projection of $Ba(MoO_2)_2(PO_4)_2$.

chains which run along the *c* axis (Fig. 5). We observe two chains situated at $x = 0.25$ and $x = 0.75$.

These chains are the same as in $Pb(MoO_2)_2(PO_4)_2$ but they are less curved than the former. Along the *a* axis run also chains of $-PO_4-MoO_6-PO_4-MoO_6-$. Thus in $Ba(MoO_2)_2(PO_4)_2$ two tunnels are built inside of which Ba atoms are situated at $x = 0.0$. In the tunnels Ba atoms have ten oxygen-atom neighbors.

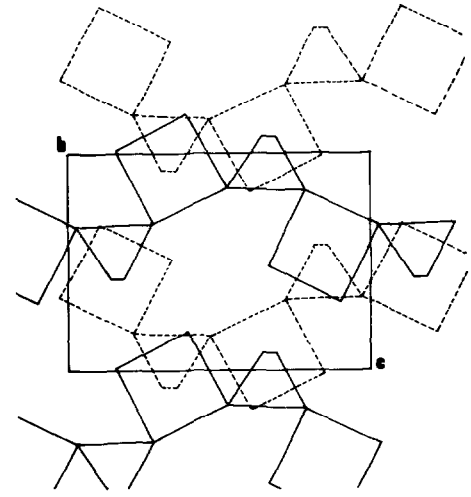

FIG. 5. Chains of octahedra and tetrahedra running along the *c* axis in $Ba(MoO_2)_2(PO_4)_2$.

TABLE X

INTERATOMIC DISTANCES (Å) AND ANGLES (°) FOR $Ba(MoO_2)_2(PO_4)_2$

	O1	O2	O3	O4
O1	1.537(3)	110°1(1)	107°7(1)	106°9(1)
O2	2.523(4)	1.529(2)	113°7(1)	110°8(1)
O3	2.480(4)	2.565(3)	1.534(2)	107°3(1)
O4	2.479(3)	2.533(3)	2.483(3)	1.549(2)

Mo–O1 = 2.065(2)	Mo–O4 = 1.958(2)
Mo–O2 = 2.217(2)	Mo–O5 = 1.695(2)
Mo–O3 = 2.130(2)	Mo–O6 = 1.703(2)
O1–Mo–O2 = 84.5(1)	O2–Mo–O6 = 89.4(1)
O1–Mo–O3 = 76.0(1)	O3–Mo–O4 = 88.2(1)
O1–Mo–O4 = 161.6(1)	O3–Mo–O5 = 91.5(1)
O1–Mo–O5 = 93.4(1)	O3–Mo–O6 = 162.9(1)
O1–Mo–O6 = 92.9(1)	O4–Mo–O5 = 96.4(1)
O2–Mo–O3 = 76.8(1)	O4–Mo–O6 = 100.1(1)
O2–Mo–O4 = 82.7(1)	O5–Mo–O6 = 102.2(1)
O2–Mo–O5 = 168.3(1)	
Ba–O1 = 3.143(3) (×2)	Ba–O5 = 2.724(2) (×2)
Ba–O2 = 2.797(2) (×2)	Ba–O6 = 2.872(3) (×2)
Ba–O3 = 2.864(3) (×2)	

Main interatomic distances and bond angles are reported in Table X. We should note the equivalence of B_{eq} (Å^2) isotropic factors in the two structures Pb$(MoO_2)_2(PO_4)_2$ and $Ba(MoO_2)_2(PO_4)_2$. Considerations about isotropic thermal factors indicate what oxygen atom is more or less bonded to the molybdenum atom.

In these two structures, $Pb(MoO_2)_2(PO_4)_2$ and $Ba(MoO_2)_2(PO_4)_2$, we have found the same atomic arrangement $\{MoO_6–PO_4–\}$ as in $NaMoO_2PO_4$ (*2*) and $AgMoO_2PO_4$ (*5*).

References

1. P. KIERKEGAARD, *Ark. Kemi,* **19,** 51 (1962).
2. P. KIERKEGAARD, *Ark. Kemi,* **18,** 553 (1962).
3. G. A. BUKHALOVA, I. V. MARDIROSOVA, AND N. P. OCHERET, *Izv. Akad. Nauk. SSSR Neorg. Mater.,* **18,** 1057 (1982).
4. Program system SDP, Version RSX19M, September 1977, Enraf-Nouius.
5. P. KIERKEGAARD AND S. HOLMEN, *Ark. Kemi,* **23,** 213 (1964).