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The Crystal Structure of $Mo(OH)_3PO_4$

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$Mo(OH)_3PO_4$ has a monoclinic unit cell containing two formula units and with the dimensions

$$a = (6.731 \pm 0.005) \text{ Å}$$
$$b = (6.319 \pm 0.005) \text{ Å}$$
$$c = (7.034 \pm 0.005) \text{ Å}$$
$$\beta = (110.16 \pm 0.05)^\circ$$
$$V = 280._8 \text{ Å}^3$$

A structure is proposed with atomic positions in the space group No. 11, $P2_1/m$. The positions of the molybdenum and phosphorus atoms in the unit cell were determined by Fourier methods. The parameters of the oxygen atoms were determined partly from electron density projections and partly from geometrical considerations, interatomic distances from previously determined structures being used. The crystals are built up of double chains parallel to the y-axis and formed by MoO_6 octahedra, coupled together by PO_4 groups so that every MoO_6 octahedron is sharing edges with three phosphate tetrahedra and every PO_4 tetrahedron with three MoO_6 octahedra. The double chains are then held together by the hydrogen atoms situated between adjacent oxygen atoms from different chains.

In connection with an X-ray investigation of oxygen-phosphorus compounds of molybdenum and wolfram undertaken at this institute a crystal structure determination of a molybdenum(VI)-phosphate, $2\ MoO_3 \cdot P_2O_5 \cdot 3\ H_2O$, has been made. The existence of a crystalline compound of this composition was reported by Schulz [1]. The crystals were described as slightly soluble in cold water but dissolving easily in warm water, giving a strongly acid solution, yellow when warm but colourless at room temperature. The compound did not lose water until it was heated to a temperature above 225°C and, as a water-free final product $2\ MoO_3 \cdot P_2O_5$ was formed. From this and some other experiments it was concluded by Schulz that the first compound was an orthophosphate with the formula $MoO_2HPO_4 \cdot H_2O$ and the second was a pyrophosphate, $(MoO_2)_2P_2O_7$. A structure determination of the latter has been started but here we shall deal only with the first-mentioned compound.

PREPARATION OF THE CRYSTALS

A viscous solution of MoO_3 (15 g) in concentrated (14.5 M) H_3PO_4 (45 ml) was prepared at about 180°C. On cooling the solution was diluted with 400 ml of concentrated (15.5 M) HNO_3 and then evaporated, during which time small crystals were obtained. The evaporation was continued to a volume of 200 ml after which the crystals were filtered off, washed with cold water and dried in air. (The method according to Schulz [1].)

The crystals were small colourless rods. However, the yield was low and the crystals obtained were too small for X-ray work and therefore the synthesis was repeated several times, resulting in sufficient amounts of the small crystals. The biggest single crystal obtained after growth in hot solution was one of about 0.1 mm in length and 0.03 mm in diameter.

Analysis. A sample was dissolved in hot water. From an ammoniacal solution the *phosphorus* was precipitated (according to Kolthoff and Sandell [2]) with a magnesium solution. The precipitate of magnesium phosphate was filtered off, washed with water, dissolved in hot 1 M HNO_3 and from this solution the phosphorus finally was precipitated with ammonium molybdate and weighed as $(NH_4)_3PO_4 \cdot 12\ MoO_3$. The amount of *molybdenum* in the filtrate was determined gravimetrically by precipitation with 8-hydroxyquinoline [3,4] from a solution slightly acid with acetic acid. The precipitate was filtered off, washed with water, dried at 140°C and weighed as molybdenum hydroxyquinolate. The *water* was determined by Hartwig-Bendig's [5] modification of Brush's [6] and Penfield's [7] methods.

The *density* of the crystals was determined from the apparent loss of weight in benzene.

	Calculated for $2\ MoO_3 \cdot P_2O_5 \cdot 3\ H_2O$	Found
% MoO_3	59.48	59.7
% P_2O_5	29.35	29.5
% H_2O	11.17	10.7
Density	2.862	2.82

The density is calculated for one formula unit per unit cell.

UNIT CELL AND SPACE GROUP

From rotation photographs (around [010] and [100]) and the corresponding Weissenberg photographs (zero, first and second layer lines), taken with CuK radiation it was concluded that the crystals are monoclinic with $a = 6.8$ Å, $b = 6.4$ Å, $c = 7.1$ Å and $\beta = 110°$ (the b-axis coincides with the needle axis of the crystal).

The reflections were recorded photographically with multiple film techniques and the relative intensities were estimated visually by comparison with an intensity scale obtained by photographing a reflection with different exposure times. However, the accuracy in the intensity estimations was rather low since the photographs were fairly poor owing to the smallness of the crystals prepared. The values of F^2 were then calculated from the relative intensities using the polarization and Lorentz' factor given by Lu [8]. No correction was applied for absorption.

More accurate values for the unit cell dimensions were calculated from a powder photograph taken with monochromatized Cu$K\alpha_1$ radiation in a focusing camera of Guinier type. KCl was used as an internal standard substance (see Table 1).

$$
\begin{aligned}
a &= (6.731 \pm 0.005)\ \text{Å}\\
b &= (6.319 \pm 0.005)\ \text{Å}\\
c &= (7.034 \pm 0.005)\ \text{Å}\\
\beta &= (110.1_6 \pm 0.05)^\circ\\
V &= 280._8\ \text{Å}^3
\end{aligned}
$$

The value (2.82) found for the density gives 0.99 ≈ 1 formula unit in the unit cell.

The reflections systematically absent are

$$0k0 \text{ with } k = \text{odd}$$

This is characteristic of the two space groups No. 4, $P2_1$, and No. 11, $P2_1/m$. The investigation was started by examining whether the structure was consistent with the latter space group, having the highest symmetry. In No. 11, $P2_1/m$, the following point positions are possible.

$2(a)$: 000; $0\frac{1}{2}0$; $2(b)$: $\frac{1}{2}00$; $\frac{1}{2}\frac{1}{2}0$; $2(c)$: $00\frac{1}{2}$; $0\frac{1}{2}\frac{1}{2}$
$2(d)$: $\frac{1}{2}0\frac{1}{2}$; $\frac{1}{2}\frac{1}{2}\frac{1}{2}$; $2(e)$: $\pm(x\frac{1}{4}z)$
$4(f)$: $\pm(xyz)$; $\pm(x,\frac{1}{2}\text{-}y,z)$

Table 1. Powder photograph of $Mo(OH)_3PO_4$. $CuK\alpha_1$-radiation. $\lambda_{CuK\alpha_1} = 1.54050$ Å.

h k l	$10^4\sin^2\Theta$ calc	$10^4\sin^2\Theta$ obs	pF^2 calc	*I* obs	*h k l*	$10^4\sin^2\Theta$ calc	$10^4\sin^2\Theta$ obs	pF^2 calc	*I* obs
0 0 1	136	136	6	w	1 1 2	1036	1033	25	w
1 0 0	149	149	4	w	2 1 1	1074	—	1	—
1 0 $\bar{1}$	187	187	62	vst	1 0 $\bar{3}$	1079	—	0	—
0 1 1	284	289	36	m	1 2 $\bar{2}$	1087	—	0	—
1 1 0	296	296	21	w	2 2 $\bar{1}$	1125	1122	15	vw
1 1 $\bar{1}$	334	334	36	m	0 2 2	1134	1134	5	vvw
1 0 1	383	382	40	st	3 0 $\bar{1}$	1180	1182	14	vw
1 0 $\bar{2}$	497	497	10	vw	2 2 0	1184		18	
1 1 1	530	530	19	w	0 0 3	1225	1222	43	m
2 0 $\bar{1}$	535	533	28	w	1 1 $\bar{3}$	1227		26	
0 0 2	544	543	16	w	2 0 $\bar{3}$	1231	—	12	—
0 2 0	590	592	85	vvst	3 0 $\bar{2}$	1294	1289	64	w
2 0 0	594		31		3 1 $\bar{1}$	1327	1323	36	vw
1 1 $\bar{2}$	644	—	0	—	2 2 $\bar{2}$	1337	1332	1	vvw
2 1 $\bar{1}$	682	681	12	vvw	3 0 0			11	
0 1 2	692	690	62	st	0 1 3	1372	1368	9	vvw
0 2 1	726	—	1	—	2 1 $\bar{3}$	1379	—	8	—
1 2 0	739	739	2	vvw	3 1 $\bar{2}$	1441	—	1	—
2 1 0	742	—	0	—	0 3 1	1464	1464	30	w
2 0 $\bar{2}$	747	744	3	vvw	1 3 0	1476	1476	25	w
1 2 $\bar{1}$	777	778	29	m	1 2 2	1479	—	1	—
1 0 2	889	891	4	vvw	3 1 0	1485	—	15	—
2 1 $\bar{2}$	894		10		1 3 $\bar{1}$	1514	1515	26	w
2 0 1	927	—	1	—	2 2 1	1516		1	
1 2 1	973	972	14	vw	2 0 2	1531	1528	14	vvw

The powder photograph was measured and interpreted to $\sin^2\Theta = 0.41$.
Reflections systematically absent in space group $P2_1/m$ have been omitted.
KCl ($a = 6.2930$ Å) has been used as an internal standard substance.

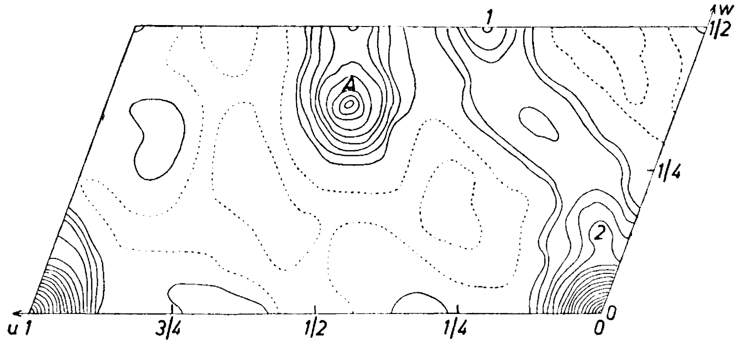

Fig. 1. The Patterson function $P(upw)$ for $Mo(OH)_3PO_4$. Dashed lines indicate negative values.

POSITIONS OF THE MOLYBDENUM AND PHOSPHORUS ATOMS

In order to find the positions of the two molybdenum atoms the Patterson projections, $P(upw)$ and $P(pvw)$, were calculated (Figs. 1 and 2). Those atoms must occupy one of the twofold positions, *i.e.* 2(a)—(e). Now, in the Patterson projections on the uw and vw plane there are high peaks (besides the origin maxima) at $u = 0.57_0$, $w = 0.36_2$ in $P(upw)$ (A in Fig. 1), and at $v = \frac{1}{2}$, $w = 0.35_2$ in $P(pvw)$ (B in Fig. 2). (All maxima have been located by the interpolation table given by Booth [9].) Thus, in the half-cell there is a high peak at $u = 0.57$, $v = \frac{1}{2}$, $w = 0.36$ and this maximum certainly corresponds to the Mo—Mo vector. The molybdenum atoms thus must occupy the point position 2(e), as the other twofold point positions would require the Mo—Mo vector to have the specialized coordinates $u = \frac{1}{2}$, $v = 0$, $w = \frac{1}{2}$. From the coordinates given above we get

$$2 \text{ Mo in } P2_1/m \ 2(e) \text{ with } x \approx 0.285, \ y = 1/4, \ z \approx 0.180$$

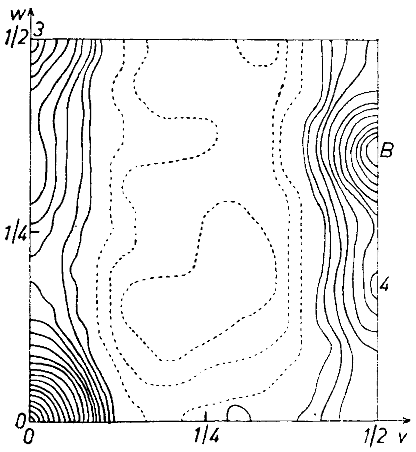

Fig. 2. The Patterson function $P(pvw)$ for $Mo(OH)_3PO_4$. Dashed lines indicate negative values.

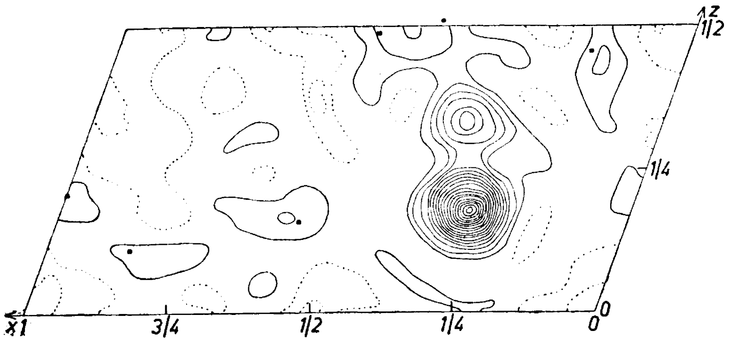

Fig. 3. Electron density of $Mo(OH)_3PO_4$ projected on the *xz* plane. The positions of the oxygen atoms O_2-O_6 (■) have been marked. Dashed lines indicate negative values.

In addition to the peak corresponding to the Mo—Mo vector the Patterson projections also show maxima, the heights of which suggest that they can be ascribed to the Mo—P distances. Thus, the peaks 1 ($u = 0.38$, $w = 0.50$) and 2 ($u = 0.06$, $w = 0.15$) in $P(upw)$ (Fig. 1) and 3 ($v = 0$, $w = 0.50$) and 4 ($v = \frac{1}{2}$, $w = 0.16$) in $P(pvw)$ (Fig. 2) define two maxima in the Patterson function, *viz.* at $u = 0.38$, $v = 0$, $w = 0.50$ and at $u = 0.06$, $v = \frac{1}{2}$, $w = 0.15$. These Mo—P vectors are only in accordance with the two phosphorus atoms occupying the point position 2(*e*) with the approximate coordinates

$$x_P = x_{Mo} + 0.38 = 0.67$$
$$y_P = 1/4$$
$$z_P = z_{Mo} + 0.50 = 0.68$$

POSITIONS OF THE OXYGEN ATOMS

Electron density projections. In order to obtain more accurate values of the molybdenum and phosphorus coordinates the electron density projec-

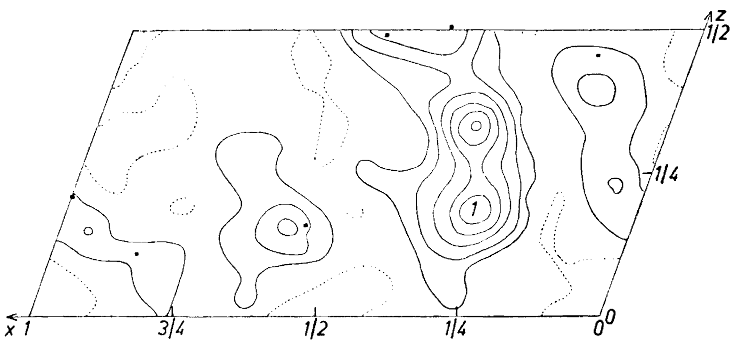

Fig. 4. Electron density of $Mo(OH)_3PO_4$ projected on the *xz* plane and with the molybdenum atom substracted. The positions of the oxygen atoms have been indicated as in Fig. 3. Dashed lines indicate negative values.

tion on the xz and yz planes were calculated. The signs of all structure factors $F(h0l)$ and $F(0kl)$ were determined from the contributions given by the molybdenum atoms. The results are given in Figs. 3 and 5.

From Fig. 3 we see that the molybdenum and the phosphorus atoms are clearly indicated at $x = 0.282$, $z = 0.176$ and at $x = 0.34$, $z = 0.34$. However, the "phosphorus" peak might be a little displaced from the correct phosphorus position by diffraction effects caused by the heavy molybdenum atom. In order to get, probably more reliable, coordinates for the phosphorus atoms the molybdenum atom was subtracted from the Fourier image in Fig. 3. — The contributions of the Mo atoms to the structure factors were multiplied by a "temperature" factor of $10^{-0.315} \sin^2\Theta$, obtained in the ordinary way for the determination of temperature factors.

From Fig. 4, which shows this new electron density projection, we see the phosphorus atom at $x = 0.32_7$, $z = 0.33_8$. However, there is yet another maximum (1 in Fig. 4, with $x = 0.28_7$, $z = 0.18_8$) with nearly the same positions as the molybdenum atom. The height of the peak is about the same as the maximum corresponding to the phosphorus position and if it is a real one the only atoms which could give a maximum of this height are two overlapping oxygen atoms, which will occur if four oxygen atoms occupy the point position 4(f). Now, from Fig. 5 showing the electron density projected on the yz plane, we see that there are two peaks (1') outside ($i.e.$ $y = 0.54$ and 0.96) the mirror planes ($y = \pm 1/4$) and with the z parameter ($z = 0.18_9$) in close agreement with the z parameter found above for peak 1 in Fig. 4. It seems therefore very probable that peak 1 is a real maximum and that four oxygen atoms are situated in the point position 4(f). (The accuracy of the x-coordinate of these oxygen atoms might be reduced owing to inadequate subtraction of the overlapping Mo atom [10].)

The molybdenum and phosphorus atoms stand out very clearly in $\varrho(pyz)$ (Fig. 5), the molybdenum at $y = 1/4$, $z = 0.177$ and the phosphorus at $y = 3/4$,

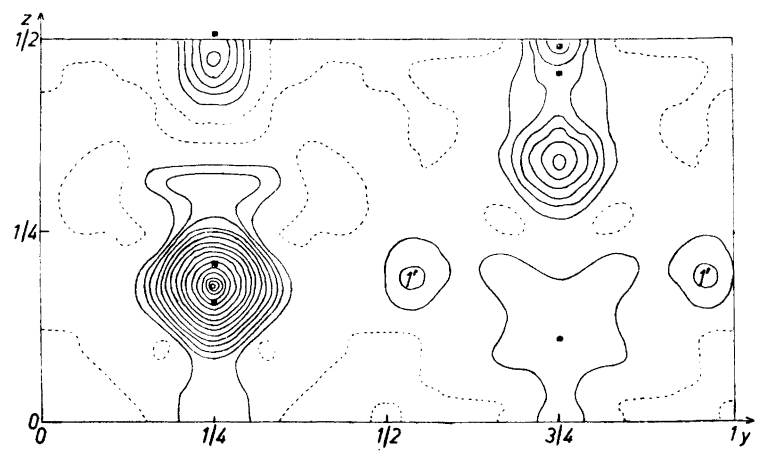

Fig. 5. Electron density of $Mo(OH)_3PO_4$ projected on the yz plane. The positions of the oxygen atoms have been indicated as in Fig. 3. Dashed lines indicate negative values.

$z = 0.34_0$, the z parameters being in good agreement with those obtained from $\varrho(xpz)$. The values obtained from $\varrho(xpz)$ should, however, be more correct, since in the calculation of this projection more F-values were used. Thus from the electron density projections the following parameters could be obtained:

2 Mo	in $P2_1/m$	2(e)	with:	$x = 0.282$,	$y = 1/4$,	$z = 0.176$
2 P	in »	»	»	$x = 0.67_3$,	$y = 1/4$,	$z = 0.66_2$
4 O_1	in »	4(f)	»	$x = 0.71_3$,	$y = 0.46_0$	$z = 0.81_1$

The remaining maxima in the electron density projections were, at this state of the investigation, not considered as they are rather diffuse and of about the same height as the false maxima [11]. However, as will be seen (see p. 1708) they were found to correspond to oxygen positions.

Geometrical considerations. The four oxygen atoms (O_1), the positions of which have been found from the electron density projections, are situated close to phosphorus atoms with the distances P—2 O_1 = 1.6_5 Å. These distances are in good agreement with P—O distances found in previously described structures [12–14] (*viz.* P—O = 1.4_5—1.6_5 Å). Assuming that the coordination figure around the phosphorus atoms is a fairly regular tetrahedron, which seems to be very probable, it is then possible to calculate the parameters of the remaining oxygen atoms (O_2 and O_3) in the PO_4 tetrahedron. If we assume the bond lengths P—O_2 = P—O_3 = 1.5_5 Å and O_2—O_3 = 2.5_3 Å (these distances are mean values of those published [12–14]) the coordinates of the oxygen atoms O_2 and O_3 follow immediately. Thus we obtain

2 O_2	in $P2_1/m$	2(e)	with:	$x = 0.44_3$,	$y = 1/4$,	$z = 0.50_9$
2 O_3	in »	»	»	$x = 0.83_0$,	$y = 1/4$,	$z = 0.54_4$

Now we have to find the positions of the remaining six oxygen atoms in the unit cell. They must occupy three of the twofold or one of the twofold and one of the fourfold positions, if the arrangement of these atoms also has the symmetry $P2_1/m$. Assuming that the distances between these oxygen atoms and the oxygens already located (*i.e.* O_1—O_3) are not less than 2.6_0 Å, the twofold positions 2(a)—2(d) can at once be eliminated and thus the remaining oxygen atoms are situated in 4 × 2(e) or 2(e) + 4(f). Of these point positions, 4(f) seems to be improbable since nothing indicates this in the electron density projections. (Figs. 3—5.) Moreover, with the oxygen parameters found above, we have three oxygen atoms (2 O_1 and O_2) in contact with one molybdenum atom (*viz.* Mo—2 O_1 = 1.8_3 Å and Mo—O_2 = 2.2_2 Å, in good agreement with Mo—O distances found in previously determined structures of oxygen compounds of molybdenum [15–18] and these four atoms are situated in the same plane. Now, if we have four oxygens in 4(f) these oxygens cannot be in contact with the molybdenum atoms since all O—O distances ought to be $\geq 2.6_0$ Å and thus we would obtain a very improbable coordination, quite different from previously known arrangements [16–18]. For these reasons we conclude that the remaining six oxygen atoms must occupy the point positions 2(e).

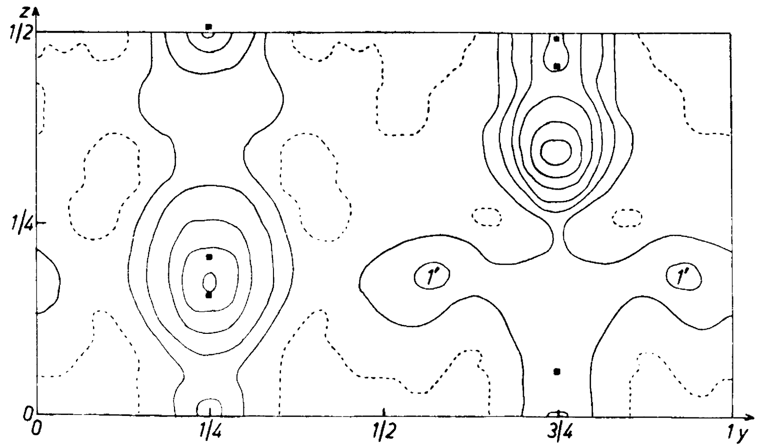

Fig. 6. Electron density of $Mo(OH)_3PO_4$ projected on the *yz* plane and with the molybdenum atom substracted. The positions of the oxygen atoms have been indicated as in Fig. 3. Dashed lines indicate negative values.

Now, with the assumption made above for the O—O distances and assuming the distance Mo—O to be not less than that found [15] in MoO_3, *i.e.* 1.8_0 Å, reasonable interatomic distances were only obtained assuming

$$2\ O_4 \text{ in } P2_1/m\ 2(e) \text{ with } x = 0.15_0,\quad y = 1/4,\quad z = 0.89_0$$
$$2\ O_5 \text{ » » » » } x = 0.57_3,\quad y = 1/4,\quad z = 0.15_8$$
$$2\ O_6 \text{ » » » » } x = 0.99_6,\quad y = 1/4,\quad z = 0.20_8$$

When the coordinates of all oxygen atoms have been determined the positions of these atoms were checked against the electron density projections (Figs. 3—5). The positions of the oxygen atoms O_2—O_6 have been denoted in these figures and, as we see, they are situated in positive areas and in most cases nearly coinciding with small peaks. However, these maxima are spread over a rather wide area, probably because of inadequate intensity material owing to the smallness of the crystal, so it is not possible to obtain any coordinates from the positions of these peaks. From Fig. 5 we see that the oxygen atoms O_5 and O_6 coincide with the molybdenum atom in this projection. In order to check if these oxygen atoms can be distinguished, the molybdenum atom was also subtracted from this Fourier image (in the same way as with Fig. 3, see p. 1706). The result is given in Fig. 6 and we see the phosphorus and oxygen (1') peaks with the same positions as in Fig. 5. The remaining maxima in Fig. 6 are well explained since they correspond to oxygen positions.

ARRANGEMENT OF THE HYDROGEN ATOMS

In the unit cell there are also six hydrogen atoms, which should be situated close to some of the oxygen atoms. Now, the oxygen atoms are not all equivalent in the sense that they have not the same environment. Thus the oxygen

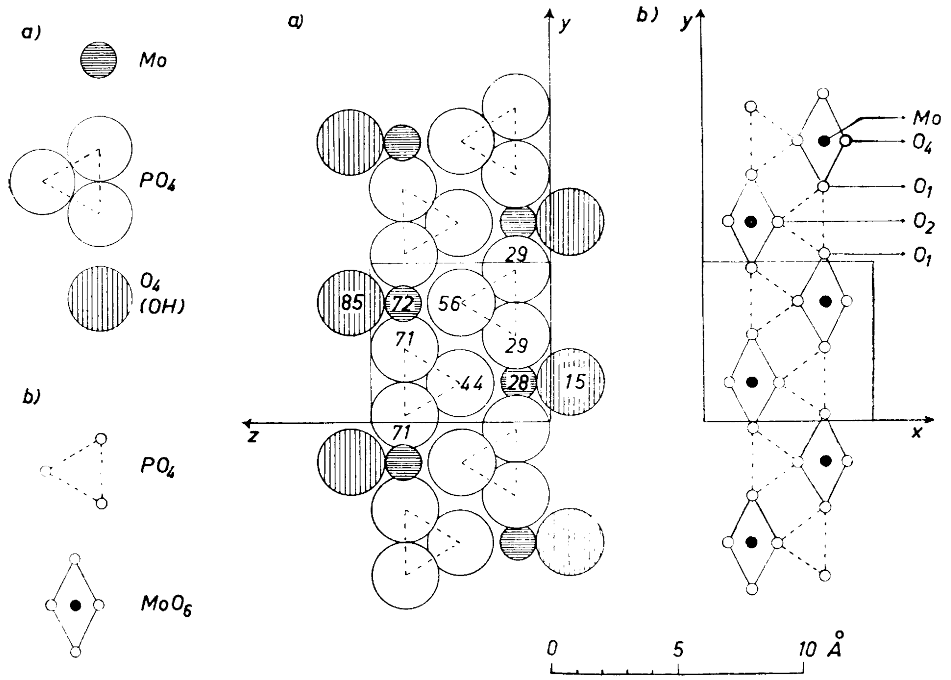

Fig. 7. The contacts between MoO_6-octahedra and PO_4-tetrahedra showing the double chains in $Mo(OH)_3PO_4$. The oxygen atoms O_5 and O_6 in the MoO_6-octahedra are situated over or below the molybdenum atoms and have not been indicated. a) Projection of the *yz* plane. The figures denote the height of the atoms in percentages of *a*. b) Projection of the *xy* plane.

atoms O_1 and O_2 are in contact with phosphorus and also with molybdenum atoms, while the oxygen atom O_3 is in contact only with one phosphorus atom and the oxygen atoms O_4—O_6 only with one molybdenum atom (*cf.* Fig. 7). It seems therefore much more probable that the hydrogen atoms are situated close to six of the oxygen atoms O_3—O_6. Now, as the proton affinity of O^{2-} is greater than that of PO_4^{3-}, it seems much more likely that the hydrogen atoms are situated close to all or some of the oxygen atoms O_4—O_6 than to the phosphate oxygen atom O_3 and some of the oxygen O_4—O_6. Thus, the six oxygen atoms O_4—O_6 exist either as six OH^- or as three O^{2-} and three H_2O, of which the first alternative seems to be the most plausible. As the interatomic distances between the oxygen atoms O_4—O_6 and neighbouring oxygen atoms are about 2.7_5 Å, the hydroxyl ions almost certainly form hydroxyl bonds (Ref.[13], p. 413) with neighbouring oxygen atoms. However, in this investigation it has not been possible to determine the parameters of the hydrogen atoms.

With the arrangement proposed for the hydrogen atoms there are no water molecules present in the crystals — which may also be supported by the fact that the crystals do not lose water until it is heated to above 225°C [1] — but they contain three hydroxyl groups for one molybdenum atom. Their formula should then be written $Mo(OH)_3PO_4$ rather than $2\ MoO_3 \cdot P_2O_5 \cdot 3\ H_2O$ or $MoO_2HPO_4H_2O$.

FINAL STRUCTURE PROPOSITION

The following structure is thus proposed for $Mo(OH)_3PO_4$. Space group: No. 11, $P2_1/m$. Two formula units per unit cell.

	x	y	z
2 Mo in 2(*e*)	0.282 ± 0.003	1/4	0.176 ± 0.003
2 P in 2(*e*)	0.673 ± 0.006	1/4	0.662 ± 0.006
4 O_1 in 4(*f*)	0.71	0.46	0.81
2 O_2 in 2(*e*)	0.44	1/4	0.51
2 O_3 in 2(*e*)	0.83	1/4	0.54
2 O_4 in 2(*e*)	0.15	1/4	0.89
2 O_5 in 2(*e*)	0.57	1/4	0.16
2 O_6 in 2(*e*)	0.00	1/4	0.21

2(*e*): $\pm(x, 1/4, z)$
4(*f*): $\pm(x,y,z)$; $\pm(x,\frac{1}{2}-y,z)$

The accuracy of the oxygen parameters is estimated to be about ±0.01. The structure factors were calculated with these parameters, multiplied by the exponential factor $10^{-0.315} \sin^2\Theta$ (*cf.* above) and compared with the observed structure factors from the corresponding Weissenberg photographs (Table 2). Taking into account that, particularly in $0kl$ and $1kl$ dependent on the shape of the crystals, there is an absorption effect [11] in the reflections, the agreement between observed and calculated intensities is quite good. The reliability index, R, valculated according to Booth (*cf.* Ref.[9], p. 101), was found to be 0.17 for $h0l$ and 0.19 for $0kl$ (absent reflections not included).

DESCRIPTION OF STRUCTURE

In the structure, the molybdenum atoms are surrounded by six oxygen atoms in an octahedral arrangement. These MoO_6 octahedra are linked together by phosphate tetrahedra so that every molybdenum atom is in contact with three PO_4 tetrahedra and every PO_4 tetrahedron with three molybdenum atoms so that the cyrstals are built up of double chains extending to [010] and containing alternately MoO_6 and PO_4 groups (*cf.* Fig. 7). These double chains with the composition $(Mo_2O_6(PO_4)_2^{6-})_n$ are then held together by the hydrogen atoms, situated between adjacent oxygen atoms (O_3—O_6) from different chains.

The xz projection of the structure is shown in Fig. 8.

The distances between adjacent atoms in the structure will be (*in* Å)

Table 2. Comparison between calculated and observed structure factors from Weissenberg photographs of $Mo(OH)_3PO_4$. Cu*K*-radiation.

h k l	*F* calc	|*F*| obs	*h k l*	*F* calc	|*F*| obs	*h k l*	*F* calc	|*F*| obs	*h k l*	*F* calc	|*F*| obs
1 0 8	−3	—	2 0 4	18	25	0 0 1	33	27	4 1 5	−7	—
0 0 8	−31	32	1 0 4	57	58	1 0 $\bar{1}$	111	114	3 1 5	22	24
1 0 $\bar{8}$	11	15	0 0 4	−19	24	2 0 $\bar{1}$	−75	91	2 1 5	−4	—
2 0 $\bar{8}$	34	35	1 0 $\bar{4}$	−49	53	3 0 $\bar{1}$	−53	25	1 1 5	−31	23
3 0 $\bar{8}$	−36	38	2 0 $\bar{4}$	25	28	4 0 $\bar{1}$	80	82	0 1 5	26	24
4 0 $\bar{8}$	−15	—	3 0 $\bar{4}$	38	35	5 0 $\bar{1}$	−2	—	1 1 $\bar{5}$	−24	25
5 0 $\bar{8}$	38	29	4 0 $\bar{4}$	−26	22	6 0 $\bar{1}$	−52	38	2 1 $\bar{5}$	35	37
6 0 $\bar{8}$	−9	—	5 0 $\bar{4}$	−5	—	7 0 $\bar{1}$	23	26	3 1 $\bar{5}$	37	29
			6 0 $\bar{4}$	37	31	8 0 $\bar{1}$	29	—	4 1 $\bar{5}$	−50	45
2 0 7	11	—	7 0 $\bar{4}$	3	—				5 1 $\bar{5}$	1	—
1 0 7	−46	36	8 0 $\bar{4}$	−36	30	8 0 0	−11	—	6 1 $\bar{5}$	37	29
0 0 7	−11	—				7 0 0	47	23	7 1 $\bar{5}$	−24	22
1 0 $\bar{7}$	51	45	6 0 3	10	21	6 0 0	1	—	8 1 $\bar{5}$	−14	14
2 0 $\bar{7}$	−15	22	5 0 3	23	34	5 0 0	−40	43			
3 0 $\bar{7}$	−46	33	4 0 3	−32	27	4 0 0	36	29	5 1 4	−24	25
4 0 $\bar{7}$	35	36	3 0 3	−17	27	3 0 0	46	47	4 1 4	23	29
5 0 $\bar{7}$	8	—	2 0 3	33	29	2 0 0	−79	104	3 1 4	19	19
6 0 $\bar{7}$	−45	33	1 0 3	28	37	1 0 0	−29	35	2 1 4	−20	37
7 0 $\bar{7}$	3	—	0 0 3	−92	92				1 1 4	−3	—
			1 0 $\bar{3}$	−7	—	1 1 8	26	17	0 1 4	65	68
4 0 6	8	—	2 0 $\bar{3}$	50	46	0 1 8	−30	26	1 1 $\bar{4}$	21	23
3 0 6	37	37	3 0 $\bar{3}$	−7	—	1 1 $\bar{8}$	8	—	2 1 $\bar{4}$	58	44
2 0 6	−23	23	4 0 $\bar{3}$	−53	54	2 1 $\bar{8}$	−29	28	3 1 $\bar{4}$	−61	51
1 0 6	−40	41	5 0 $\bar{3}$	19	13	3 1 $\bar{8}$	−13	18	4 1 $\bar{4}$	−21	13
0 0 6	59	51	6 0 $\bar{3}$	40	25	4 1 $\bar{8}$	20	20	5 1 $\bar{4}$	34	28
1 0 $\bar{6}$	−4	—	7 0 $\bar{3}$	−49	36	5 1 $\bar{8}$	−5	—	6 1 $\bar{4}$	−2	—
2 0 $\bar{6}$	−55	40	8 0 $\bar{3}$	−9	—	6 1 $\bar{8}$	−15	13	7 1 $\bar{4}$	−39	29
3 0 $\bar{6}$	24	28							8 1 $\bar{4}$	17	—
4 0 $\bar{6}$	42	36	7 0 2	−6	—	2 1 7	23	29			
5 0 $\bar{6}$	−26	28	6 0 2	29	31	1 1 7	−4	—	6 1 3	−25	26
6 0 $\bar{6}$	−29	23	5 0 2	−3	—	0 1 7	−25	26	5 1 3	10	—
7 0 $\bar{6}$	29	20	4 0 2	−53	49	1 1 $\bar{7}$	−9	—	4 1 3	42	39
8 0 $\bar{6}$	−1	—	3 0 2	0	—	2 1 $\bar{7}$	−18	23	3 1 3	−34	38
			2 0 2	52	61	3 1 $\bar{7}$	23	26	2 1 3	−39	34
5 0 5	−12	—	1 0 2	−29	20	4 1 $\bar{7}$	7	—	1 1 3	67	68
4 0 5	55	37	0 0 2	−56	76	5 1 $\bar{7}$	−15	22	0 1 3	30	28
3 0 5	0	—	1 0 $\bar{2}$	44	43	6 1 $\bar{7}$	−17	14	1 1 $\bar{3}$	51	55
2 0 5	−46	38	2 0 $\bar{2}$	23	37	7 1 $\bar{7}$	26	28	2 1 $\bar{3}$	−29	17
1 0 5	35	34	3 0 $\bar{2}$	−113	131				3 1 $\bar{3}$	−41	32
0 0 5	30	36	4 0 $\bar{2}$	2	25	3 1 6	20	20	4 1 $\bar{3}$	45	35
1 0 $\bar{5}$	−42	34	5 0 $\bar{2}$	67	66	2 1 6	13	17	5 1 $\bar{3}$	14	20
2 0 $\bar{5}$	−6	—	6 0 $\bar{2}$	−25	25	1 1 6	−23	19	6 1 $\bar{3}$	−48	32
3 0 $\bar{5}$	66	56	7 0 $\bar{2}$	−40	33	0 1 6	−20	24	7 1 $\bar{3}$	−6	—
4 0 $\bar{5}$	−5	—	8 0 $\bar{2}$	25	21	1 1 $\bar{6}$	−13	—	8 1 $\bar{3}$	18	16
5 0 $\bar{5}$	−33	24				2 1 $\bar{6}$	−1	—			
6 0 $\bar{5}$	13	—	7 0 1	16	19	3 1 $\bar{6}$	31	36	7 1 2	−26	14
7 0 $\bar{5}$	24	27	6 0 1	24	29	4 1 $\bar{6}$	−6	—	6 1 2	−8	—
8 0 $\bar{5}$	−8	—	5 0 1	−28	27	5 1 $\bar{6}$	−32	37	5 1 2	33	46
			4 0 1	−37	35	6 1 $\bar{6}$	28	27	4 1 2	−8	—
5 0 4	30	26	3 0 1	75	72	7 1 $\bar{6}$	26	22	3 1 2	−69	69
4 0 4	20	15	2 0 1	13	—				2 1 2	41	58
3 0 4	−53	52	1 0 1	−89	97	5 1 5	−13	—	1 1 2	50	37

Table 2.
(cont.)

h k l	F calc	|F| obs	h k l	F calc	|F| obs	h k l	F calc	|F| obs	h k l	F calc	|F| obs
0 1 2	−79	85	0 5 1	−22	27	1 3 7	1	—	1 3 3	−66	61
1 1 $\bar{2}$	0	—	0 6 1	−9	—	1 2 7	35	35	1 2 3	−10	—
2 1 $\bar{2}$	−32	29	0 7 1	29	16	1 2 $\bar{7}$	−36	31	1 2 $\bar{3}$	1	—
3 1 $\bar{2}$	7	—	0 8 1	6	—	1 3 $\bar{7}$	7	—	1 3 $\bar{3}$	−58	54
4 1 $\bar{2}$	44	34				1 4 $\bar{7}$	39	28	1 4 $\bar{3}$	−4	—
5 1 $\bar{2}$	−20	22	0 2 2	22	27				1 5 $\bar{3}$	32	38
6 1 $\bar{2}$	−16	21	0 3 2	71	75	1 5 6	−15	15	1 6 $\bar{3}$	2	—
7 1 $\bar{2}$	18	21	0 4 2	−33	28	1 4 6	−29	31	1 7 $\bar{3}$	−37	36
8 1 $\bar{2}$	9	22	0 5 2	−42	35	1 3 6	24	29			
			0 6 2	22	16	1 2 6	26	36	1 7 2	−29	20
7 1 1	−34	27	0 7 2	40	30	1 2 $\bar{6}$	12	—	1 6 2	9	—
6 1 1	33	37				1 3 $\bar{6}$	18	23	1 5 2	25	22
5 1 1	24	21	0 2 3	51	50	1 4 $\bar{6}$	−4	—	1 4 2	−15	18
4 1 1	−59	52	0 3 3	−27	31	1 5 $\bar{6}$	−8	—	1 3 2	−50	49
3 1 1	7	—	0 4 3	−53	40				1 2 2	7	—
2 1 1	12	20	0 5 3	14	—	1 6 5	−22	25	1 2 $\bar{2}$	−7	—
1 1 1	−44	52	0 6 3	35	28	1 5 5	−19	—	1 3 $\bar{2}$	−9	—
0 1 1	−60	60	0 7 3	−14	—	1 4 5	28	32	1 4 $\bar{2}$	25	18
1 1 $\bar{1}$	−60	62				1 3 5	35	33	1 5 $\bar{2}$	−2	—
2 1 $\bar{1}$	−33	35	0 2 4	18	13	1 2 5	31	44	1 6 $\bar{2}$	−16	17
3 1 $\bar{1}$	60	61	0 3 4	−66	49	1 2 $\bar{5}$	29	31	1 7 $\bar{2}$	−6	—
4 1 $\bar{1}$	19	21	0 4 4	−15	—	1 3 $\bar{5}$	30	38			
			0 5 4	40	37	1 4 $\bar{5}$	−32	31	1 7 1	20	21
5 1 $\bar{1}$	−5	—	0 6 4	12	—	1 5 $\bar{5}$	−15	—	1 6 1	34	43
6 1 $\bar{1}$	−11	—	0 7 4	−40	23	1 6 $\bar{5}$	24	26	1 5 1	−24	33
7 1 $\bar{1}$	28	29							1 4 1	−50	47
8 1 $\bar{1}$	−7	—	0 2 5	−11	—	1 7 4	3	—	1 3 1	36	40
			0 3 5	−26	—	1 6 4	−28	25	1 2 1	37	47
7 1 0	15	—	0 4 5	21	13	1 5 4	−1	—	1 2 $\bar{1}$	−54	69
6 1 0	35	30	0 5 5	17	—	1 4 4	40	35	1 3 $\bar{1}$	51	62
5 1 0	−30	50	0 6 5	14	—	1 3 4	5	—	1 4 $\bar{1}$	59	49
4 1 0	−19	19				1 2 4	−33	37	1 5 $\bar{1}$	−27	26
3 1 0	39	45	0 2 6	−45	45	1 2 $\bar{4}$	21	32	1 6 $\bar{1}$	−40	44
2 1 0	6	—	0 3 6	22	—	1 3 $\bar{4}$	−21	22	1 7 $\bar{1}$	27	19
1 1 0	−46	46	0 4 6	46	39	1 4 $\bar{4}$	−31	22			
0 2 0	−130	187	0 5 6	−11	—	1 5 $\bar{4}$	14	—	1 8 0	−8	—
0 4 0	103	120				1 6 $\bar{4}$	20	24	1 7 0	27	34
0 6 0	−68	53	0 2 7	6	—	1 7 $\bar{4}$	−13	—	1 6 0	12	—
0 8 0	45	23	0 3 7	28	30				1 5 0	−19	21
			0 4 7	−8	—	1 7 3	−40	27	1 4 0	−17	26
0 2 1	−8	13				1 6 3	−9	—	1 3 0	50	54
0 3 1	55	66	0 2 8	20	16	1 5 3	41	41	1 2 0	15	16
0 4 1	14	—	1 2 $\bar{8}$	−8	—	1 4 3	16	—			

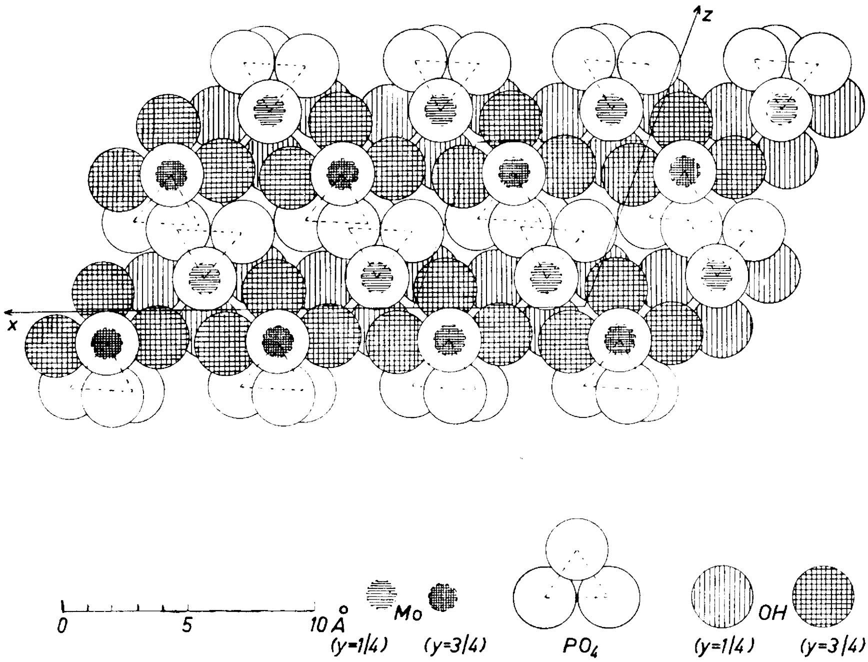

Fig. 8. Projection on the *xz* plane of the structure of $Mo(OH)_3PO_4$.

Mo—2 O_1;	O_1—Mo	= 1.8_3
Mo—O_2 ;	O_2—Mo	= 2.2_2
Mo—O_4 ;	O_4—Mo	= 1.9_0
Mo—O_5 ;	O_5—Mo	= 2.0_0
Mo—O_6 ;	O_6—Mo	= 2.0_1
P —2 O_1;	O_1—P	= 1.6_5
P —O_2 ;	O_2—P	= 1.5_5
P —O_3 ;	O_3—P	= 1.5_5
O_1 —O_1		= 2.6_5
O_1 —2 O_2;	O_2—4 O_1	= 2.7_0; 2.8_1
O_1 —O_3 ;	O_3—2 O_1	= 2.6_3
O_1 =2 O_4;	O_4—4 O_1	= 2.7_0; 3.1_0
O_1—O_5;	O_5—2 O_1	= 2.7_2
O_1—2 O_6;	O_6—4 O_1	= 2.7_2; 3.1_0
O_2—2 O_2		= 3.2_6
O_2—O_3;	O_3—O_2	= 2.5_3
O_2—O_5;	O_5—O_2	= 2.8_9
O_2—O_6;	O_6—O_2	= 3.0_2
O_3—O_4;	O_4—O_3	= 2.6_4
O_3—O_5;	O_5—O_3	= 2.6_7
O_3—O_6;	O_6—O_3	= 3.0_8
O_4—O_5;	O_5—O_4	= 2.8_2
O_4—2 0_6;	O_6— $2O_4$	= 2.7_7; 3.4_5
O_5—O_6;	O_6—O_5	= 2.7_4

The O—O distances are all within the normal range, showing that the lattice is also supported by O—O contacts. However, the distances may be in error by a few tenths of an Ångström unit, because the oxygen positions could not be determined very accurately.

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