Multicomponent Polyanions. 44. The Structure of Tetraguanidinium Dihydrogenpentamolybdodiphosphate Monohydrate

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The crystal structure of $[C(NH_2)_3]_4H_2Mo_5P_2O_{23} \cdot H_2O$ was determined from X-ray single-crystal data. Anisotropic refinement in the space group $P2_1/c$ resulted in $R = 0.043$, using 7011 unique reflections. Cell parameters are $a = 14.608(2)$, $b = 13.054(2)$, $c = 15.887(2)$ Å and $\beta = 92.47(1)°$. The structure contains $H_2Mo_5P_2O_{23}{}^{4-}$ anions, $C(NH_2)_3{}^+$ cations and water molecules. The links between these units are mainly due to hydrogen bonds. The anions are arranged in layers almost parallel to (100).

In our department the aqueous molybdophosphate system has been extensively studied since the late 1960s. The aim has been to determine the composition, formation constants and structures of complexes formed. A variety of experimental techniques has been used such as potentiometry, spectroscopy (NMR, IR, Raman) and X-ray diffraction on single crystals and concentrated aqueous solutions.[1-6] The equilibria have been written with H^+, $MoO_4{}^{2-}$ and $HPO_4{}^{2-}$ as components. Thus the complexes are formed according to reaction (1), where p, q and r are

$$pH^+ + qMoO_4^{2-} + rHPO_4^{2-} \rightleftharpoons (H^+)_p(MoO_4^{2-})_q(HPO_4^{2-})_r \quad (1)$$

integers. For brevity the complexes are often given the notation (p,q,r), and homonuclear series are referred to by their (q,r) values. The equilibrium investigations give the compositions (the p, q and r values) and the formation constants, $\beta_{p,q,r}$, of complexes formed. However, no information about their structures is obtained. In order to obtain structural information, crystallisation experiments on solutions with known composition have been performed. The complexity of the molybdophosphate system was clearly shown in combined EMF–^{31}P NMR studies.[1,2] With two exceptions the structures of species formed were known. For the isomeric (9,1) species an α-B structure[7a] was proposed, and the (11,1) species were thought to have a lacunary α-Keggin structure.[7a] Complementary crystallisation experiments using a variety of cations were performed to prove these proposed structures. Since the (9,1) and (11,1) phases were the ones of interest, solutions with rather high Mo/P ratios were used. The crystallisation experiments were mostly performed on Mo/P = 7–9 solutions in which the (9,1) and (11,1) complexes are the predominant ones and the (5,2) complexes are only minor

species. This is clearly illustrated in the distribution diagram shown in Fig. 1 (Mo/P = 7.5). The distribution of complexes has been calculated using the formation constants from Ref. 2 [0.6 M Na(Cl), 25 °C]. The presence of (5,2) complexes can be further suppressed by using even higher Mo/P ratios. However, from such solutions isopolymolybdate phases will often precipitate.

The single crystal used in the present study was faintly yellow and was obtained from a Mo/P = 7.5 solution (ca. pH 4) to which some guanidinium chloride had been added. From our experience, the (5,2) complexes are colourless in aqueous solution and in the solid phase when sodium is the counter-ion. This led us to believe that the
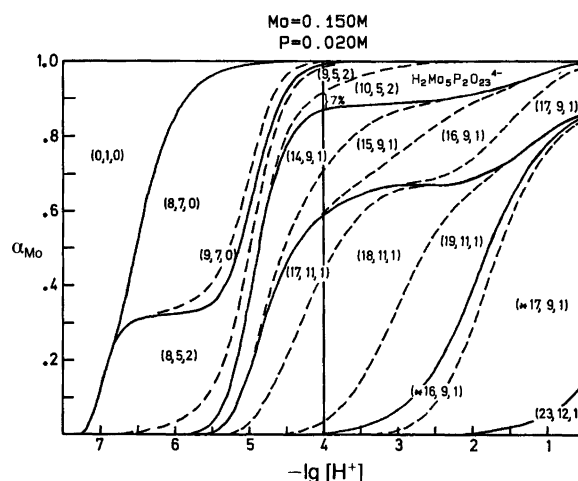

Fig. 1. Distribution diagram showing all Mo-containing species as a function of $-\lg [H^+]$ at Mo/P = 7.5. Each line represents the cumulative fractional contribution α_{Mo} of each species to the total molybdenum content. The species are denoted by their (p,q,r) values. The species within homonuclear (q,r) series are separated by dashed lines. The distribution has been calculated using the stability constants from Ref. 2.

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crystal would not contain a (5,2) complex. Moreover, a comparison of the IR spectrum with earlier recorded spectra of the sodium salts of (8,5,2), (9,5,2) and (10,5,2) complexes exposed some discrepancies that could not be attributed to the different cations. An X-ray diffraction study was therefore performed.

The X-ray structure determination showed the anions to have the well known Mo_5P_2 structure,[7b] with a composition corresponding to a (10,5,2) complex. The structure of a sodium salt $(Na_4H_2Mo_5P_2O_{23} \cdot 10H_2O)$ containing such anions has previously been published.[8] Although the guanidinium cation often behaves unpredictably in crystallisation experiments, it is somewhat surprising that a salt is precipitated with an anion which in the equilibrated aqueous solution only contains 7% of the total molybdenum. When using tetramethylammonium at the same $Mo/P = 7.5$ ratio and at the same pH an (11,1) phase has been obtained. This compound is presently the subject of a single-crystal X-ray study.

Experimental

As mentioned above, the crystals were obtained from a solution having a Mo/P ratio of 7.5. The aqueous solution (ca. pH 4) with the composition 200 mM Na_2MoO_4, 26.7 mM Na_2HPO_4, 338 mM HCl and 67 mM $C(NH_2)_3Cl$ (deficiency of guanidinium chloride) was allowed to evaporate at 25 °C. The distribution of complexes in the solution prior to precipitation is similar to that shown in Fig. 1 at ca. pH 4. After a few days faintly yellow prismatic crystals appeared from which one was selected for the crystal study. The recipe used was aimed at obtaining crystals of the (9,1) or (11,1) compounds and was thus not optimized for the (10,5,2) complex. The latter is the predominant one at significantly lower Mo/P ratios.

The intensities of the reflections were measured with a Syntex R3 X-ray diffractometer (graphite-monochromated $MoK\alpha$ radiation). The background of the reflections was measured on each side of the peak for a total time equal to the scan time. In addition to Lorentz and polarization corrections, an empirical absorption correction was applied. Twenty reflections, evenly distributed in the measured 2Θ range, were selected. Each reflection was rotated around its diffraction vector in steps of 10°, resulting in a transmission factor variation of 0.795–1.000.

The structure was solved using Patterson synthesis and standard heavy-atom methods. No extinction correction was necessary. Scattering factors were used for Mo^{3+}, P, O^-, N and C. The anomalous dispersion was taken into account.[9] Attempts to locate the H atoms were not made.

Computer programs described by Antti[10] and programs supplied with the Syntex R3 crystallographic system were used. Computations were performed with a CDC Cyber 850/180 computer at the University Computer Centre and a Data General Nova 3 computer.

Crystal data and further data collection details are given in Table 1, and refinement results in Table 2.

Table 1. Crystal and experimental data for $[C(NH_2)_3]_4H_2Mo_5P_2O_{23} \cdot H_2O$ at $T = 298$ K.

M_r	1170.0
Crystal system	Monoclinic
Space group	$P2_1/c$ (No. 14)
$a/Å$	14.608(2)
$b/Å$	13.054(2)
$c/Å$	15.887(2)
$\beta/°$	92.47(1)
$V/Å^3$	3026.7(7)
Z	4
$D_c/g\ cm^{-3}$	2.57
$\mu(MoK\alpha)/mm^{-1}$	2.18
Crystal size/mm	$0.18 \times 0.12 \times 0.07$
No. of reflections for cell determination (Θ range/°)	25 (9 < Θ < 12.5)
Scan mode	$\Theta-2\Theta$
2Θ range/°	4.6 < 2Θ < 65.0
2Θ scan speed/° min^{-1}	2.0–6.0
Total no. of reflections measured	11816
No. of observed independent reflections [$I > 3\sigma(I)$]	7011
Test reflections (standard deviation/%)	$0\ \bar{4}\ 0$ (1.4); $3\ 2\ \bar{3}$ (2.3); $2\ 4\ \bar{4}$ (1.6); $\bar{6}\ 2\ 6$ (1.5)
No. of parameters refined	443
Weights calculated according to $R\ (R_w)$	$w = [\sigma^2(F_0) + (0.0154F_0)^2]^{-1}$ 0.043 (0.044)
Maximum residual electron density/e $Å^{-3}$	1.30

Description and discussion

The structure consists of $H_2Mo_5P_2O_{23}^{4-}$ anions, $C(NH_2)_3^+$ cations and water molecules. The anions, oriented with their pentagon of Mo atoms almost parallel to (100), are connected by numerous hydrogen bonds involving guanidinium ions as well as water molecules (Fig. 2).

The $H_2Mo_5P_2O_{23}^{4-}$ anion. The structure of the anion is built up of five MoO_6 octahedra and two PO_4 tetrahedra (Fig. 3). The octahedra form a ring by sharing four edges and one corner. To each side of the ring a tetrahedron is attached which has three oxygens in common with the MoO_6 octahedra. The H atoms are considered to be attached to the unshared phosphate oxygen atoms (see discussion below).

Selected distances within the anion are given in Table 3. When the MoO_6 octahedra share edges the Mo–Mo distances vary between 3.377(1) and 3.403(1) Å, while when corner-sharing the distance increases to 3.706(1) Å. As can be seen from Table 3, the octahedra are far from being regular, and the Mo–O distance increases with the number of atoms coordinating the O atom. Each octahedron comprises two terminal O atoms, two O atoms being shared by two Mo, and two O atoms shared between P and one or two Mo. Consequently, three different groups of Mo–O bonds are present with the following distances: 1.693(4)–1.714(5), 1.907(4)–1.946(4) and 2.217(4)–2.466(4) Å.

Table 2. Fractional atomic coordinates, occupancy factors not equal to unity and B_{eq} values (Ref. 16) with e.s.d.s in parentheses.[a]

Atom	x	y	z	Occ.	$B_{eq}/\text{Å}^2$
Mo1	0.26463(3)	0.24758(4)	0.38337(3)		1.22(1)
Mo2	0.25438(3)	0.23480(3)	0.59668(3)		1.26(1)
Mo3	0.21340(3)	0.47259(4)	0.66980(3)		1.28(1)
Mo4	0.25999(3)	0.64834(4)	0.49353(3)		1.40(1)
Mo5	0.26472(3)	0.49529(4)	0.32151(3)		1.47(1)
P1	0.37826(9)	0.42735(10)	0.51387(9)		1.16(3)
P2	0.12122(9)	0.42105(10)	0.47554(8)		1.10(3)
O(12)	0.3122(3)	0.2083(3)	0.4927(2)		1.45(8)
O(15)	0.2170(2)	0.3590(3)	0.3174(2)		1.39(8)
O(23)	0.1889(2)	0.3277(3)	0.6670(2)		1.46(8)
O(34)	0.2342(3)	0.5912(3)	0.6006(2)		1.62(9)
O(45)	0.3022(3)	0.6180(3)	0.3834(2)		1.78(9)
OP1	0.4705(3)	0.3715(3)	0.5391(3)		1.73(9)
OP2	0.0330(3)	0.4131(3)	0.4161(3)		1.83(9)
OP(12)	0.1646(2)	0.3148(3)	0.4792(2)		1.33(8)
OP(15)	0.3479(3)	0.3979(3)	0.4234(2)		1.43(8)
OP(23)	0.3086(2)	0.3912(3)	0.5770(2)		1.26(8)
OP(3)	0.0957(2)	0.4565(3)	0.5619(2)		1.28(8)
OP(4)	0.3892(3)	0.5428(3)	0.5222(3)		1.65(9)
OP(45)	0.1858(3)	0.5011(3)	0.4379(2)		1.46(8)
O1(1)	0.1842(3)	0.1546(3)	0.3641(3)		1.99(10)
O2(1)	0.3563(3)	0.2105(3)	0.3286(3)		1.91(9)
O1(2)	0.1768(3)	0.1366(3)	0.5976(3)		2.36(11)
O2(2)	0.3425(3)	0.1948(4)	0.6614(3)		2.24(10)
O1(3)	0.1246(3)	0.5143(3)	0.7271(3)		2.09(10)
O2(3)	0.3050(3)	0.4837(3)	0.7381(3)		2.12(10)
O1(4)	0.1594(3)	0.7116(3)	0.4694(3)		2.42(11)
O2(4)	0.3328(3)	0.7446(3)	0.5215(3)		2.49(11)
O1(5)	0.1837(4)	0.5500(4)	0.2563(3)		2.94(12)
O2(5)	0.3590(3)	0.4865(4)	0.2628(3)		2.54(11)
C1	0.4652(4)	0.9326(5)	0.5893(4)		2.37(15)
C2	0.4236(5)	0.7208(5)	0.7410(4)		2.40(15)
C3	0.0424(5)	0.9039(6)	0.5735(5)		2.81(17)
C4	0.0266(11)	0.7513(14)	0.7663(9)	0.72(2)	2.57(29)
C4'	0.0315(24)	0.7458(41)	0.7968(21)	0.28(2)	2.44(73)
N1(1)	0.5181(4)	0.9682(5)	0.6533(4)		3.37(16)
N2(1)	0.4985(4)	0.8702(5)	0.5317(4)		2.80(14)
N3(1)	0.3776(4)	0.9630(5)	0.5817(4)		3.17(15)
N1(2)	0.4703(5)	0.7728(6)	0.7998(4)		4.28(19)
N2(2)	0.4658(4)	0.6579(5)	0.6882(4)		3.07(15)
N3(2)	0.3321(4)	0.7301(5)	0.7354(5)		3.62(17)
N1(3)	0.0024(4)	0.8243(5)	0.5378(5)		4.08(19)
N2(3)	−0.0016(5)	0.9649(6)	0.6250(4)		4.53(21)
N3(3)	0.1287(4)	0.9265(5)	0.5557(4)		3.64(17)
N1(4)	−0.0352(4)	0.6832(6)	0.7545(4)		3.59(17)
N2(4)	0.0147(5)	0.8256(6)	0.8270(5)		4.92(22)
N3(4)	0.1065(6)	0.7466(7)	0.7265(8)	0.72(2)	3.89(31)
N3(4')	0.1207(12)	0.7124(17)	0.7967(15)	0.28(2)	2.47(55)
O(aq)	0.2619(5)	0.9451(5)	0.4154(4)		5.29(20)

[a]For the O atoms O(i), O(ij) and OP(ij), the index means that the atom is bonded to Mo atoms i (and j) and P indicates that it is also bonded to the P atom. For the N atoms N(i) the index means that the atom is bonded to C atoms i.

The tetrahedral coordination around phosphorus is almost regular, with bond distances P–O 1.510(4)–1.569(4) Å and O–P–O angles 106.1(2)–111.7(2)°. It is of particular interest that the two distances to unshared oxygen atoms are the longest ones, 1.568(4) and 1.569(4) Å; undoubtedly a result of the attachment of H atoms. This is in accordance with earlier findings concerning (5,2) complexes; bonds between P and terminal O having a proton attached are significantly longer than bonds between P and Mo-shared O.[8,11,12] When the terminal O is not protonated, the bond is in the 1.49–1.51 Å range.[11-14] Apparently, the hydrogen atom gives rise ot a lengthening of the P–O bond by about 0.07 Å. The presence of peaks in the difference Fourier map in appropriate positions close to the terminal oxygens supports the presence of these hydrogens.
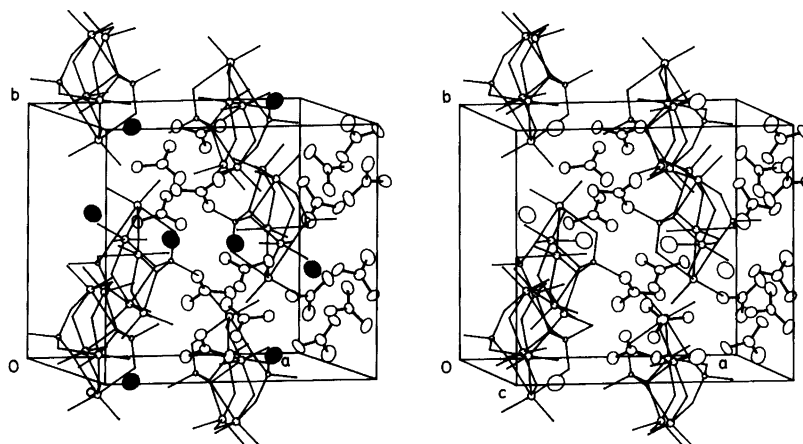
Fig. 2. A stereoscopic view of the unit cell. The water oxygens [O(aq)] are shaded.

Table 3. Selected distances (in Å) and O–Mo–O angles (in °) within the $H_2Mo_5P_2O_{23}{}^{4-}$ anion.

Mo1–Mo2	3.403(1)	P1–Mo1	3.502(1)	P2–Mo1	3.453(1)
Mo1–Mo5	3.380(1)	P1–Mo2	3.395(1)	P2–Mo2	3.616(1)
Mo2–Mo3	3.377(1)	P1–Mo3	3.578(1)	P2–Mo3	3.381(1)
Mo3–Mo4	3.706(1)	P1–Mo4	3.371(1)	P2–Mo4	3.598(1)
Mo4–Mo5	3.388(1)	P1–Mo5	3.527(1)	P2–Mo5	3.430(1)

	O1(1)	O2(1)	O(12)	O(15)	OP(12)	OP(15)
Mo1	1.708(4)	1.699(4)	1.913(4)	1.907(4)	2.326(4)	2.381(4)
O1(1)–Mo1–		104.9(2)	101.1(2)	102.2(2)	86.5(2)	
OP(15)–Mo1–		88.1(2)	79.5(1)	71.9(1)	81.0(1)	
O2(1)–Mo1–			97.2(2)	102.3(2)		
OP(12)–Mo1–			73.8(2)	81.2(1)		

	O1(2)	O2(2)	O(12)	O(23)	OP(12)	OP(23)
Mo2	1.711(4)	1.695(4)	1.918(4)	1.930(4)	2.466(4)	2.217(4)
O1(2)–Mo2–		104.5(2)	100.8(2)	97.0 2)	89.6(2)	
OP(23)–Mo2–		95.9(2)	82.6(2)	72.0(2)	71.6(1)	
O2(2)–Mo2–			96.7(2)	102.9(2)		
OP(12)–Mo2–			70.4(1)	85.0(1)		

	O1(3)	O2(3)	O(23)	O(34)	OP(3)	OP(23)
Mo3	1.706(4)	1.693(4)	1.925(4)	1.931(4)	2.384(4)	2.326(4)
O1(3)–Mo3–		103.2(2)	100.4(2)	100.9(2)	82.5(2)	
OP(23)–Mo3–		88.2(2)	69.7(1)	83.8(2)	86.4(1)	
O2(3)–Mo3–			103.9(2)	99.0(2)		
OP(3)–Mo3–			76.7(1)	77.8(2)		

	O1(4)	O2(4)	O(34)	O(45)	OP(45)	OP(4)
Mo4	1.714(5)	1.693(4)	1.909(4)	1.921(4)	2.359(4)	2.365(4)
O1(4)–Mo4–		102.9(2)	100.8(2)	101.6(2)	86.2(2)	
OP(4)–Mo4–		83.9(2)	77.7(2)	76.8(1)	87.0(1)	
O2(4)–Mo4–			101.6(2)	99.8(2)		
OP(45)–Mo4–			84.9(2)	69.6(2)		

	O1(5)	O2(5)	O(15)	O(45)	OP(45)	OP(15)
Mo5	1.696(5)	1.700(4)	1.912(4)	1.946(4)	2.222(4)	2.354(4)
O1(5)–Mo5–		104.7(2)	97.3(2)	97.8(2)	96.9(2)	
OP(15)–Mo5–		86.2(2)	72.4(1)	88.4(2)	73.6(1)	
O2(5)–Mo5–			102.8(2)	96.6(2)		
OP(45)–Mo5–			81.9(2)	72.3(2)		
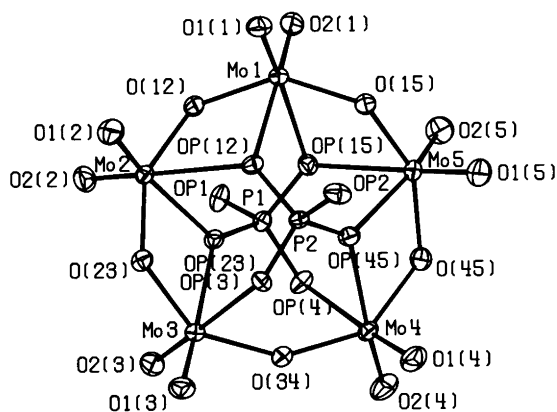
Fig. 3. The $Mo_5P_2O_{23}$ unit. The thermal ellipsoids are scaled to include 50 % probability (Ref. 17).

Table 4. Distances (in Å) and angles (in °) within the $C(NH_2)_3^+$ cations.

C1-N1(1)	1.33(1)	N1(1)-C1-N2(1)	121.4(6)
C1-N2(1)	1.33(1)	N1(1)-C1-N3(1)	119.0(6)
C1-N3(1)	1.34(1)	N2(1)-C1-N3(1)	119.6(6)
C2-N1(2)	1.32(1)	N1(2)-C2-N2(2)	121.3(6)
C2-N2(2)	1.34(1)	N1(2)-C2-N3(2)	118.9(6)
C2-N3(2)	1.34(1)	N2(2)-C2-N3(2)	119.8(6)
C3-N1(3)	1.31(1)	N1(3)-C3-N2(3)	121.6(7)
C3-N2(3)	1.33(1)	N1(3)-C3-N3(3)	119.4(7)
C3-N3(3)	1.34(1)	N2(3)-C3-N3(3)	119.0(7)
C4-N1(4)	1.27(2)	N1(4)-C4-N2(4)	118.8(13)
C4-N2(4)	1.38(2)	N1(4)-C4-N3(4)	121.3(14)
C4-N3(4)	1.35(2)	N2(4)-C4-N3(4)	119.6(14)
C4'-N1(4)	1.42(4)	N1(4)-C4'-N2(4)	123.5(30)
C4'-N2(4)	1.18(5)	N1(4)-C4'-N3(4')	116.6(36)
C4'-N3(4')	1.37(4)	N2(4)-C4'-N3(4')	119.8(34)

The $C(NH_2)_3^+$ cations. The C and N atoms in each guanidinium ion have a planar configuration, with C being triangularly coordinated by N. The C–N distances vary between 1.31(1) and 1.34(1) Å and the N–C–N angles fall in the 118.9(6)–121.6(7)° range, as shown in Table 4. The guanidinium(4) ion is, however, an exception to this pattern. In the preliminary refinements one nitrogen in the guanidinium(4) ion [with its carbon labelled C(4)] showed an abnormally large cigar-shaped thermal ellipsoid. An attempt to refine this nitrogen based upon a partial occupation of two positions was found to be successful. This indicated that the guanidinium(4) ion is disordered with respect to two slightly different orientations, (4) and (4'). A Hamilton test[15] on the final disordered guanidinium(4) model showed this disorder to be significant as compared to

the results based upon a model with a single non-disordered guanidinium(4) ion.

The difference between the two orientations of the guanidinium(4) ion has its origin in the hydrogen bonds to N3(4). The N–O distances indicate that in one case N3(4) is hydrogen-bonded to O1(5) and in the other to O1(4). This results in two different positions of both C4 and N3(4), while the positions of N1(4) and N2(4) are essentially the same.

These two guanidinium(4) orientations can be described as a rotational displacement by 34.6° around an axis going through the positions of N1(4) and N2(4) (Fig. 4). In the final refinement the occupancies of C4 and C4' were constrained to the occupancies of N3(4) and N3(4'), respectively. The occupancy factor ratio between guanidinium(4) and (4') was found to be ca. 2.5.
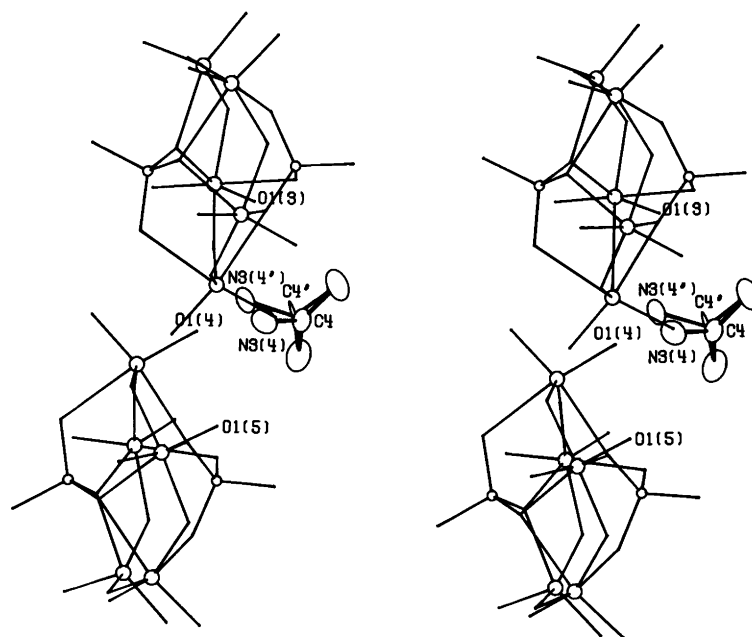

Fig. 4. A stereoscopic view of the two guanidinium(4) and (4') orientations.

Hydrogen bonds. The only links between the $H_2Mo_5P_2O_{23}^{4-}$ anions are hydrogen bonds effected by the guanidinium ions, the water molecule and the two anion-hydrogens. Although the H atoms were not definitely located, a proposal for the hydrogen-bond system can be given taking into consideration N–O and O–O distances as well as O–N–O and O–O(aq)–O angles. The system is rather complicated, and only the most obvious connections will be considered here.

As mentioned above, the $H_2Mo_5P_2O_{23}^{4-}$ anions form layers almost parallel to (100) (Fig. 2). Within and between these layers the anions are connected through hydrogen bonds, mainly by the guanidinium ions, forming O$\cdots$H–N–H$\cdots$O bridges. The N–O distances in these links are in the range 2.902(8)–3.209(8) Å and with O–N–O angles varying between 107.5(2) and 154.9(3)°.

In addition to filling up a vacancy in the structure, the water molecule exerts a bridging function within the layers through hydrogen bonds. The water molecule is hydrogen-bonded to two terminal oxygen atoms from different anions with O(aq)–O1(1) and O(aq)–O2(3) distances of 3.059(8) and 3.056(8) Å. Furthermore, the water molecule is hydrogen-bonded to two separate guanidinium ions with O(aq)–N3(1) and O(aq)–N3(3) distances of 3.083(9) and 3.031 (9) Å, respectively. This coordination can schematically be described in the following way:

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O···H \                 H–N
        \   . ·      . ·
          O(aq)
        /        . ·
O···H  /            ·  H–N
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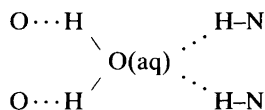
The two hydrogen atoms in the anion also take part in the hydrogen-bond system. They form strong hydrogen bonds between the anion layers, OP1–OP(4) and OP2–OP(3), with distances of 2.564(5) and 2.572(5) Å, respectively.

References

1. Pettersson, L., Andersson, I. and Öhman, L.-O. *Acta Chem. Scand., Ser. A 39* (1985) 53.
2. Pettersson, L., Andersson, I. and Öhman, L.-O. *Inorg. Chem. 25* (1986) 4726.
3. Lyhamn, L. *Chem. Scr. 12* (1977) 153.
4. Lyhamn, L. and Pettersson, L. *Chem. Scr. 12* (1977) 142.
5. Lyhamn, L. and Pettersson, L. *Chem. Scr. 16* (1980) 52.
6. Johansson, G., Pettersson, L. and Ingri, N. *Acta Chem. Scand., Ser. A 28* (1974) 1119.
7. Pope, M. T. *Heteropoly and Isopoly Oxometalates*, Springer-Verlag, Berlin 1983, (a) p. 59; (b) pp. 28–29.
8. Hedman, B. *Acta Chem. Scand. 27* (1973) 3335.
9. *International Tables for X-Ray Crystallography*, Kynoch Press, Birmingham 1974, Vol. IV.
10. Antti, B.-M. *Acta Chem. Scand., Ser. A 30* (1976) 24.
11. Hedman, B. and Strandberg, R. *Acta Crystallogr., Sect. B 35* (1979) 278.
12. Fischer, J., Ricard, L. and Toledano, P. J. *Chem. Soc., Dalton Trans.* (1974) 941.
13. Strandberg, R. *Acta Chem. Scand. 27* (1973) 1004.
14. Hedman, B. *Acta Crystallogr., Sect. B 33* (1977) 3083.
15. Hamilton, W. C. *Acta Crystallogr. 18* (1965) 502.
16. Hamilton, W. C. *Acta Crystallogr. 12* (1959) 609.
17. Johnson, C. K. *ORTEP II*, Report ORNL-5138, Oak Ridge National Laboratory, Oak Ridge, TN 1976.