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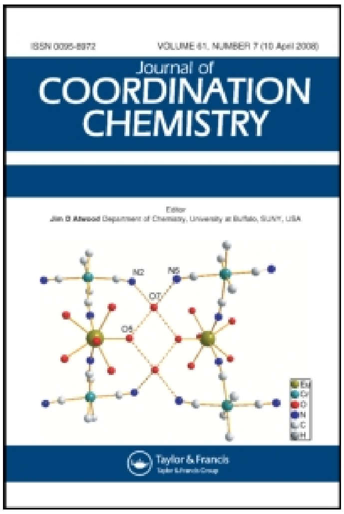

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Microwave synthesis, structure, and properties of molybdophosphate compounds

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Microwave synthesis, structure, and properties of molybdophosphate compounds

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Two compounds of disphosphopentamolybdate(VI) with the formulae $(C_4H_{14}N_2O)_3$-$[P_2Mo_5O_{23}]\cdot 3H_2O$ (**1**) and $(C_4H_{14}N_2O)_5(NH_4)_2[P_2Mo_5O_{23}]_2\cdot 5H_2O$ (**2**) have been synthesized by a simple, fast and efficient microwave method, and their crystal structures and spectroscopic properties have been studied. The heteropoly anion $[P_2Mo_5O_{23}]^{6-}$ has a known structure built of five MoO_6 octahedra and two PO_4 tetrahedra. The MoO_6 octahedra form a pentagonal ring by sharing four edges and one corner and the PO_4 tetrahedra are attached to one side of the ring by three oxygen atoms. The most interesting feature of compound **1** is its extensive framework based on a one-dimensional zigzag chain. In compound **2**, a more extensive and complicated framework of hydrogen bonds joins the anions, organic cations and water molecules together.

Keywords: Supermolecular compound; Phosphopolyoxometallate complex; Molybdenum complex; Crystal structure; Microwave synthesis

1. Introduction

Supramolecular compounds of polyoxometalates produced by combining different inorganic and organic moieties by means of molecular assemblies, are of current interest in some important areas of chemistry such as catalysis, non-linear optical materials, liquid crystals and charge-transfer salts [1–3]. The phosphomolybdates constitute a particularly well-researched group of heteropolyoxometalates. For example, compounds with Mo : P ratios ranging from 2.5 to 12 have been characterized in the solid state and/or shown to be present in aqueous solution [4, 5]. For the diphosphopentamolybdate anion, $[P_2Mo_5O_{23}]^{6-}$, crystal structures have been reported for two sodium salts, $Na_6[P_2Mo_5O_{23}]\cdot 13H_2O$ and $Na_6[P_2Mo_5O_{23}]\cdot 14H_2O$ [6, 7]. Protonation of the phosphomolybdate anion is possible as seen in, for example, $Na_4[H_2P_2Mo_5O_{23}]\cdot 10H_2O$ [8], $(NH_4)_5[HP_2Mo_5O_{23}]\cdot 3H_2O$ [9], $K_4[H_2P_2Mo_5O_{23}]\cdot 2H_2O$ [10] and $(NH_4)_8Ni(HPO_4)_2(PO_4)_2Mo_{10}O_{30}\cdot 12H_2O$ [11]. Recently, a series of extensive framework supramolecular compounds based on diphosphopentamolybdate cluster $[H_xP_2Mo_5O_{23}]^{(6-x)-}$ $(x = 0, 1, 2)$ through the linking

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of organoamine bases has been reported, such as $(C_5H_7N_2)_6[P_2Mo_5O_{23}] \cdot 5H_2O$ [12], $(C_2H_{10}N_2)_3[P_2Mo_5O_{23}] \cdot 6H_2O$ [12], $(C_4H_{12}N)_4[H_2P_2Mo_5O_{23}] \cdot 5H_2O$ [12], $(bipyH_2)_2[H_2P_2Mo_5O_{23}] \cdot H_2O$ [13], $[NH_3(CH_2)_6NH_3]_7[NH_3(CH_2)_2NH_3]_2(H_3O)_6[P_2Mo_5O_{23}]_4 \cdot 11H_2O$ [14], $(C_6H_{18}N_2)_3[P_2Mo_5O_{23}] \cdot 4H_2O$ [15], and $(C_6H_{14}N_2)_5[HP_2Mo_5O_{23}] \cdot 4H_2O$ [16]. The choice of the organoamine bases was determined by the presence of anion groups capable of establishing strong hydrogen bonds which involve the oxygen atoms as acceptors and stabilize the crystal structure of this kind of compound. Furthermore, it is well known that organic cation features, such as length, size and number of amino groups, play an import role in the crystal packing of polyoxometalate salts [12].

Among the above successful syntheses, however, most are obtained from hydrothermal conditions. In this article, we report two new compounds of disphosphopentamolybdate $(C_4H_{14}N_2O)_3[P_2Mo_5O_{23}] \cdot 3H_2O$ (**1**) and $(C_4H_{14}N_2O)_5(NH_4)_2[P_2Mo_5O_{23}]_2 \cdot 5H_2O$ (**2**) by a simple, fast and efficient microwave method. Their crystal structures and properties were studied.

2. Experimental

2.1. *Synthesis*

2.1.1. $(C_4H_{14}N_2O)_3[P_2Mo_5O_{23}] \cdot 3H_2O$ (1). To an aqueous solution (50 mL) of $Na_2MoO_4 \cdot 2H_2O$ (0.74 g, 3 mmol), and $Na_2HPO_4 \cdot 12H_2O$ (0.36 g, 1 mmol), *N*-(2-hydroxyethyl)ethylenediamine (0.31 g, 3 mmol) was added. The final pH of the solution was adjusted to 6 by using HCl (1.0 M). The mixture was placed in the cavity of a microwave synthesizer. After irradiation at 285 W for 5 min, the reaction mixture was cooled to room temperature. The solution was filtered and the filtrate was allowed to stand at room temperature for one day, giving crystals suitable for X-ray diffraction. Yield: 85%. Anal. Calcd for **1** (%): C, 9.36; N, 6.55; H, 3.74; Found: C, 9.19; N, 6.61; H, 3.78.

2.1.2. $(C_4H_{14}N_2O)_5(NH_4)_2[P_2Mo_5O_{23}]_2 \cdot 5H_2O$ (2). This compound was prepared by the same method as for **1**, but with $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ instead of $Na_2MoO_4 \cdot 2H_2O$. Yield: 76%. Anal. Calcd for **2** (%): C, 9.69; N, 6.78; H, 3.55; Found: C, 9.78; N, 6.85; H, 3.41.

2.2. *Physical measurements*

Elemental analyses (C, N and H) were carried out with a Vario EL elemental analyzer. IR spectra were recorded on a Bruker Tensor27 FT-IR spectrophotometer as KBr pellets. Electronic spectra were recorded on a Shimadzu 240 spectrophotometer.

2.3. *Crystal structure determination*

Intensity data for single crystals of compounds **1** and **2** were collected on a Bruker Smart-1000 CCD diffractometer with graphite-monochromated Mo-Kα radiation

($\lambda = 0.71073$ Å) at 298(2) K. The structure was solved by direct methods and refined by the full-matrix least-squares method on F^2 with anisotropic thermal parameters for all nonhydrogen atoms. The hydrogen atoms attached to water molecules and the protonated ammonium cations were determined from difference Fourier syntheses, and the other hydrogen atoms were located geometrically and refined isotropically. The data collection and refinement details are summarized in table 1.

3. Results and discussion

3.1. *Crystal structure*

The molecular structures of compounds **1** and **2** are illustrated in figures 1 and 2 with the atom numbering scheme, respectively. Selected bond distances for compound **1** and **2** are given in tables 2 and 3, respectively.

The units of the compounds contain three *N*-(2-hydroxyethyl)ethylenediammonium cations, three lattice water molecules and one diphosphopentamolybdate anion for compound **1**, and five *N*-(2-hydroxyethyl)ethylenediammonium cations, two ammonium anions, five water molecules and two diphosphopentamolybdate(VI) anions for compound **2**. The geometry of each anion is similar to that reported for $[P_2Mo_5O_{23}]^{6-}$ [6, 7, 11, 12], which is built up of five MoO_6 octahedra and two PO_4 tetrahedra. The octahedra form a pentagonal ring by sharing four edges and one corner. The two PO_4 tetrahedra are attached to the ring, one above and the other below, each having three oxygen atoms in common with the ring. Each octahedron has four bridging oxygen atoms and two terminal oxygen atoms. The Mo–O bond lengths can be classified into three groups: (a) short, Mo–O$_t$, terminal oxygen atoms, 1.692–1.768 Å; (b) medium, Mo–O$_b$, oxygen atoms being shared by two molybdenum atoms,

Table 1. Crystal data and structure refinements for compounds **1** and **2**.

Compounds	**1**	**2**
Empirical formula	$C_{12}H_{48}Mo_5N_6O_{29}P_2$	$C_{20}H_{88}Mo_{10}N_{12}O_{56}P_4$
Formula weight	1282.20	2476.30
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
a (Å)	10.371(13)	15.156(3)
b (Å)	11.083(14)	15.675(3)
c (Å)	17.76(2)	16.784(3)
α (°)	78.451(17)	63.091(2)
β (°)	75.839(17)	77.966(2)
γ (°)	79.285(17)	79.038(2)
V (Å^3)	1919(4)	3456.4(11)
Z	2	2
D_c (Mg m^{-3})	2.219	2.379
Absorption coefficient (mm^{-1})	1.776	1.966
Reflection collected	9095	22,507
Independent reflection	6390 ($R_{int} = 0.0602$)	15,923 ($R_{int} = 0.0238$)
Reflections with [$I > 2\sigma(I)$]	4524	13,084
Goodness-of-fit on F^2	1.012	1.010
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0769$, $wR_2 = 0.1706$	$R_1 = 0.0437$, $wR_2 = 0.1150$
R indices (all data)	$R_1 = 0.1067$, $wR_2 = 0.1853$	$R_1 = 0.0554$, $wR_2 = 0.1243$
Largest diff. peak and hole (e Å^{-3})	2.115 and −1.833	2.374 and −1.334

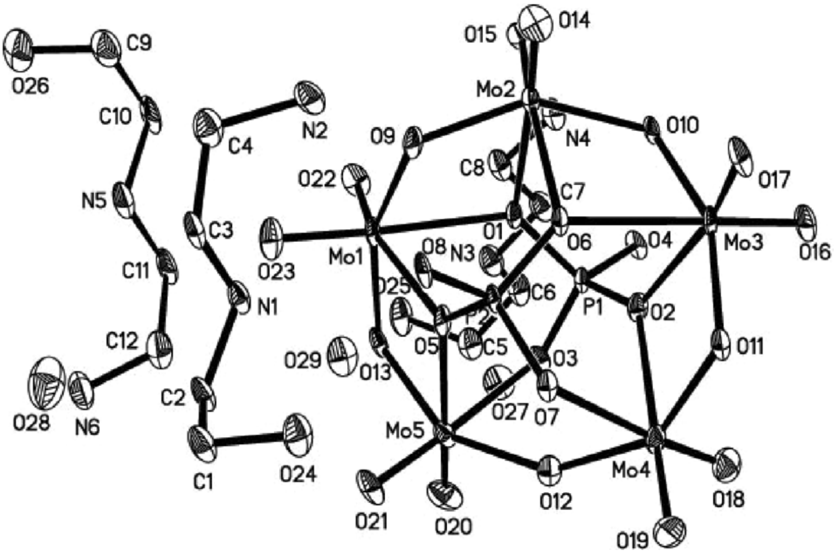

Figure 1. Molecular structure of compound **1**.

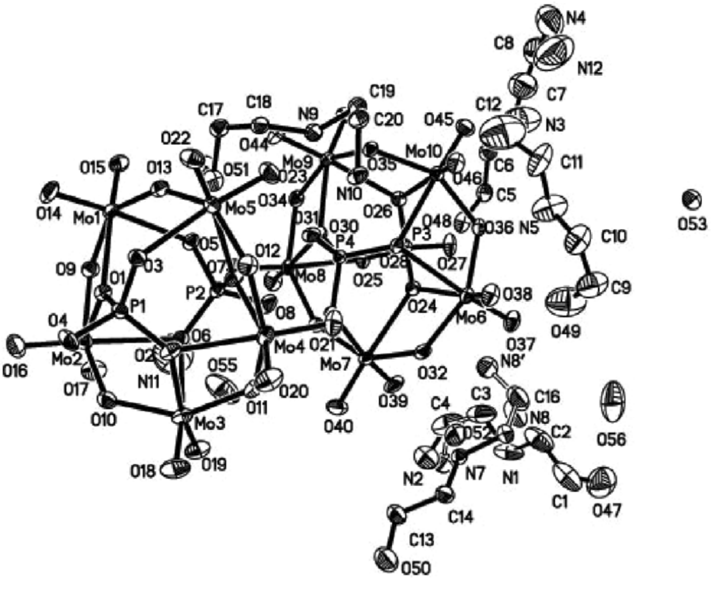

Figure 2. Molecular structure of compound **2**.

1.888–1.993 Å; (c) long, Mo–O_b, oxygen atoms being shared by phosphorus and molybdenum atoms, 2.174–2.464 Å. The Mo–O distance increases considerably as the sharing of oxygen atom increases in accord with crystal structures previously reported [12–16].

Polyanions of **1** and **2** have been compared with other organic and inorganic diphosphopentamolybdate salts previously published (table 4). The molybdenum atoms of the pentagonal ring are nearly on the same plane and no significant deviations

Table 2. Selected bond distances (Å) for compound **1**.

Mo(1)–O(22)	1.736(8)	Mo(3)–O(10)	1.983(7)
Mo(1)–O(23)	1.748(9)	Mo(3)–O(2)	2.270(7)
Mo(1)–O(9)	1.939(8)	Mo(3)–O(6)	2.324(8)
Mo(1)–O(13)	1.992(8)	Mo(4)–O(19)	1.730(9)
Mo(1)–O(5)	2.228(7)	Mo(4)–O(18)	1.734(9)
Mo(1)–O(1)	2.331(8)	Mo(4)–O(11)	1.927(7)
Mo(2)–O(14)	1.722(8)	Mo(4)–O(12)	1.973(8)
Mo(2)–O(15)	1.768(8)	Mo(4)–O(7)	2.284(8)
Mo(2)–O(9)	1.940(8)	Mo(4)–O(2)	2.430(8)
Mo(2)–O(10)	1.950(8)	Mo(5)–O(21)	1.741(8)
Mo(2)–O(1)	2.355(7)	Mo(5)–O(20)	1.743(8)
Mo(2)–O(6)	2.374(8)	Mo(5)–O(12)	1.925(8)
Mo(3)–O(17)	1.729(8)	Mo(5)–O(13)	1.948(7)
Mo(3)–O(16)	1.743(8)	Mo(5)–O(3)	2.331(8)
Mo(3)–O(11)	1.964(8)	Mo(5)–O(5)	2.339(7)
O(1)–P(1)	1.576(7)	O(5)–P(2)	1.605(7)
O(2)–P(1)	1.578(7)	O(6)–P(2)	1.542(8)
O(3)–P(1)	1.547(8)	O(7)–P(2)	1.538(7)
O(4)–P(1)	1.541(8)	O(8)–P(2)	1.544(7)

are observed as a consequence of the protonation degree. However, the influence of protonation is clearly visible in the P···P distance. This fact is clearly observed in the sodium salts for which the P···P distances range from 3.824 to 3.707 Å in non-protonated and diprotonated anions, respectively (table 4) [6, 8].

Although, the crystal packing for compounds **1** and **2** shows some common basic features with other previously reported organoammonium cations [12–16], the most notable being the extensive hydrogen bonding framework, several significant differences in compounds **1** and **2** should be noticed.

In compound **1**, three *N*-(2-hydroxyethyl)ethylenediammonium cations are diprotonated and have +2 charge, which play a different role in the formation of the framework. Two-thirds of the *N*-(2-hydroxyethyl)ethylenediammonium cations in **1** form hydrogen bonds with the terminal and bridging oxygen atoms of the $[P_2Mo_5O_{23}]^{6-}$ anion. Thus, the adjacent $[P_2Mo_5O_{23}]^{6-}$ anions are connected together to give a one-dimensional zigzag chain as shown in figure 3. One-third of cations are located between the zigzag chains and form hydrogen bonds with near lattice water molecules and the terminal oxygen atoms of the $[P_2Mo_5O_{23}]^{6-}$ anion. At the same time, the interchain multipoint hydrogen bonds are formed between lattice water molecules and the terminal and bridging oxygen atoms of the $[P_2Mo_5O_{23}]^{6-}$ anion. Therefore, the discrete $[P_2Mo_5O_{23}]^{6-}$ anions are extended into a framework through hydrogen bonds of type N–H···O (2.723–3.321 Å), N–H···Ow (2.822–3.010 Å), Ohydrox–H···O (2.759–3.080 Å), Ow–H···O (2.722–3.071 Å) and Ow–H···Ow (2.821–3.168 Å).

In compound **2**, ammonium cations also participate in the formation of a more extensive and complicated framework of hydrogen bonds of types N–H···O (2.713–3.539 Å), N–H···Ow (2.616 Å), N–H···Ohydrox (2.762 Å) N–H···N (2.763–3.852 Å), Ohydrox–H···O (2.853–3.240 Å), Ow–H···Ohydrox (2.848–2.876 Å) and Ow–H···Ow (2.779 Å) (figure 4).

Table 3. Selected bond distances (Å) for compound **2**.

Mo(1)–O(15)	1.699(4)	Mo(3)–O(11)	1.923(4)
Mo(1)–O(14)	1.712(4)	Mo(3)–O(2)	2.321(4)
Mo(1)–O(13)	1.908(4)	Mo(3)–O(6)	2.302(4)
Mo(1)–O(9)	1.918(4)	Mo(4)–O(21)	1.705(5)
Mo(1)–O(5)	2.298(4)	Mo(4)–O(20)	1.717(4)
Mo(1)–O(1)	2.406(3)	Mo(4)–O(11)	1.945(4)
Mo(2)–O(17)	1.708(4)	Mo(4)–O(12)	1.938(4)
Mo(2)–O(16)	1.695(5)	Mo(4)–O(7)	2.234(4)
Mo(2)–O(9)	1.931(4)	Mo(4)–O(2)	2.301(4)
Mo(2)–O(10)	1.926(4)	Mo(5)–O(22)	1.718(4)
Mo(2)–O(1)	2.215(4)	Mo(5)–O(23)	1.713(4)
Mo(2)–O(6)	2.421(4)	Mo(5)–O(13)	1.888(4)
Mo(3)–O(18)	1.710(4)	Mo(5)–O(12)	1.907(4)
Mo(3)–O(19)	1.709(4)	Mo(5)–O(3)	2.287(4)
Mo(3)–O(10)	1.911(4)	Mo(5)–O(7)	2.375(4)
O(1)–P(1)	1.538(4)	O(5)–P(2)	1.510(4)
O(2)–P(1)	1.548(4)	O(6)–P(2)	1.529(4)
O(3)–P(1)	1.524(4)	O(7)–P(2)	1.541(4)
O(4)–P(1)	1.535(4)	O(8)–P(2)	1.572(4)
Mo(6)–O(37)	1.692(4)	Mo(8)–O(34)	1.954(4)
Mo(6)–O(38)	1.712(4)	Mo(8)–O(25)	2.311(4)
Mo(6)–O(32)	1.936(4)	Mo(8)–O(30)	2.338(4)
Mo(6)–O(36)	1.936(4)	Mo(9)–O(43)	1.697(4)
Mo(6)–O(24)	2.174(4)	Mo(9)–O(44)	1.715(4)
Mo(6)–O(28)	2.464(4)	Mo(9)–O(34)	1.932(4)
Mo(7)–O(40)	1.692(4)	Mo(9)–O(35)	1.947(4)
Mo(7)–O(39)	1.721(4)	Mo(9)–O(30)	2.225(4)
Mo(7)–O(33)	1.918(4)	Mo(9)–O(26)	2.321(4)
Mo(7)–O(32)	1.928(4)	Mo(10)–O(46)	1.709(4)
Mo(7)–O(29)	2.234(4)	Mo(10)–O(45)	1.713(4)
Mo(7)–O(24)	2.398(4)	Mo(10)–O(36)	1.906(4)
Mo(8)–O(42)	1.701(4)	Mo(10)–O(35)	1.912(4)
Mo(8)–O(41)	1.709(4)	Mo(10)–O(26)	2.307(4)
Mo(8)–O(33)	1.892(4)	Mo(10)–O(28)	2.351(4)
O(24)–P(3)	1.545(4)	O(28)–P(4)	1.548(4)
O(25)–P(3)	1.517(4)	O(29)–P(4)	1.527(4)
O(26)–P(3)	1.549(4)	O(30)–P(4)	1.554(4)
O(27)–P(3)	1.530(4)	O(31)–P(4)	1.525(4)

Table 4. Distance from the respective ring plane of molybdenum and phosphorus atoms (Å).

Compound	Mo_1	Mo_2	Mo_3	Mo_4	Mo_5	P_1	P_2	P···P	Ref.
$(C_5H_7N_2)_6[P_2Mo_5O_{23}] \cdot 5H_2O$	0.025	−0.189	0.264	−0.250	0.150	−1.887	1.904	3.791	[12]
$(C_2H_{10}N_2)_3[P_2Mo_5O_{23}] \cdot 6H_2O$	0.045	0.115	−0.213	0.238	−0.184	1.907	−1.886	3.793	[12]
$Na_6[P_2Mo_5O_{23}] \cdot 13H_2O$	0.000	0.153	−0.232	0.232	−0.153	−1.918	1.904	3.824	[6]
$Na_4[H_2P_2Mo_5O_{23}] \cdot 10H_2O$	−0.002	0.165	−0.248	0.248	−0.163	−1.844	1.862	3.707	[8]
$(C_6H_{18}N_2)_3[P_2Mo_5O_{23}] \cdot 4H_2O$	−0.202	0.087	0.075	−0.209	0.248	1.920	−1.877	3.798	–
$(C_4H_{14}N_2O)_5(NH_4)_2$	0.2594	−0.1929	0.0352	0.1363	−0.2379	−1.9048	1.9080	3.813	–
$[P_2Mo_5O_{23}]_2 \cdot 5H_2O$	0.2039[a]	−0.2602	0.2276	−0.1187	−0.0526	1.8864	−1.9337	3.821	

–: This work.

[a] Values of Mo_6, Mo_7, Mo_8, Mo_9, Mo_{10} respectively.

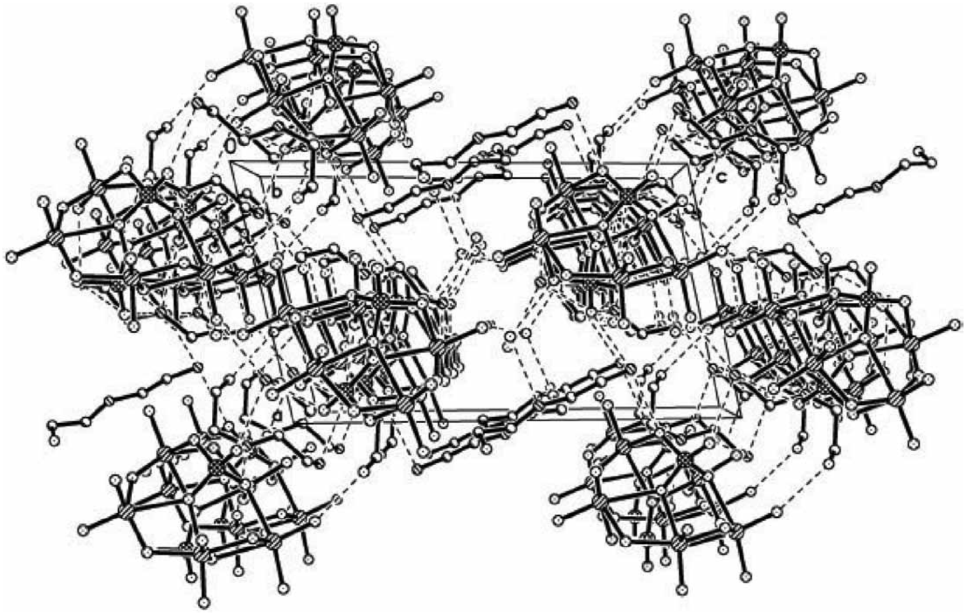

Figure 3. Packing diagram of compound **1**.

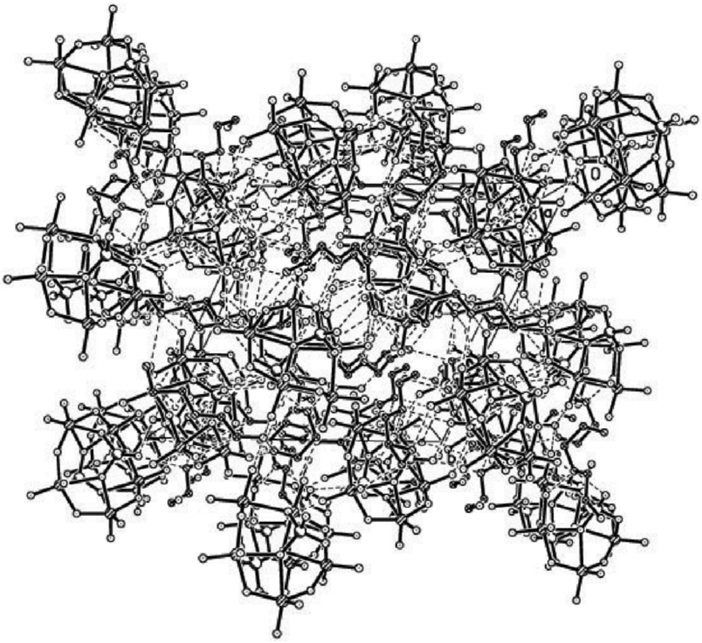

Figure 4. Packing diagram of compound **2**.

3.2. *IR spectra and electronic spectra*

IR spectra of **1** and **2** are similar and show two strong bands at ca1080 and 910 cm^{-1}, corresponding to ν(P–O) and ν(Mo–O_t), respectively. Two strong bands at ca 880 and 870 cm^{-1} are attributed to ν(Mo–O_b) [12, 13]. The bands at 3140 cm^{-1} are due to protonated N–H stretches [14]. The presence of water molecules is confirmed by absorption between 3215 and 3453 cm^{-1}, indicating intermolecular hydrogen bonds [15].

The electronic spectra of two compounds in water show two bands at ca 225 and 265 nm, which are assigned to $O_t \rightarrow Mo$ and $O_b \rightarrow Mo$ charge transfer, respectively [13, 17].

Supplementary material

Full details have been deposited with the Cambridge Crystallographic Data Centre, CCDC 606997 for **1** and 606998 for **2**. Copies of this information can be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: 44-1223-336-033; Email: deposit@ccdc.cam.ac.uk; or http://www.ccdc.cam.ac.uk).

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