

Synthesis and Crystal Structure of a Copper(II) Inorganic-Organic Hybrid Polymer Based on $[\beta\text{-Mo}_8\text{O}_{26}]^{4-}$ and 4,4'-Bipyridine-*N,N'*-dioxide

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A polyoxometalate-based copper(II) inorganic-organic hybrid polymer $[\text{Cu}(\text{dpdo})_2(\text{H}_2\text{O})_4][\text{Na}_2(\text{Mo}_8\text{O}_{26})\cdot(\text{H}_2\text{O})_6]\cdot(\text{dpdo})\cdot 2\text{H}_2\text{O}$ (**1**) (dpdo = 4,4'-bipyridine-*N,N'*-dioxide) has been synthesized and structurally characterized by IR spectroscopy, elemental analysis, and single-crystal X-ray analysis. The structure is based on two types of polymeric chains, the anionic one constructed by the coordination interaction between the $[\beta\text{-Mo}_8\text{O}_{26}]^{4-}$ cluster anions and $[\text{Na}_2]^{2+}$ units, and the cationic one formed through the coordination interaction of Cu(II) atoms and dpdo ligands. In addition, there are multiform hydrogen bonds and $\pi\cdots\pi$ interactions in a three-dimensional supramolecular network.

Key words: β -Octamolybdate, Copper(II), Polymer, Crystal Structure

Introduction

In polyoxometalate (POM) chemistry, much interest at present is focused on the design and synthesis of POM-based inorganic-organic hybrid compounds not only for their intriguing structure motifs, but also for their potential applications in the fields of catalysis, electrochemistry, magnetism, and functional materials [1–8]. Octamolybdate $[\text{Mo}_8\text{O}_{26}]^{4-}$, as an important category of polymolybdate clusters, constitutes a variety of inorganic-organic hybrid compounds due to its various isomeric forms α , β , γ , δ , ϵ , ζ , ξ , η [9]. Among them, $[\beta\text{-Mo}_8\text{O}_{26}]^{4-}$ is an outstanding inorganic building block owing to its accessibility and stability for constructing interesting POM-based inorganic-organic hybrid compounds by the introduction of N-containing ligands such as 1,3-bis(imidazol-1-yl)methylbenzene [10], benzimidazole [11], 4,4'-bipyridine [12], *N*-(2-(nicotinamido)ethyl)nicotinamide [13], 1,4-bis(imidazole-1-ylmethyl)benzene [14], or 1,4-bis(1,2,4-triazol-1-ylmethyl)-benzene [15, 16].

4,4'-Bipyridine-*N,N'*-dioxide (dpdo) is a neutral bridging ligand for the construction of inorganic-organic hybrid frameworks because of its small size

that avoids crowding at the metal centers and encourages high connectivity. Some POM-based hybrid compounds have been reported by the incorporation of metal-dpdo systems and Keggin-type heteropolyanions such as $[\text{PW}_{12}\text{O}_{40}]^{3-}$, $[\text{PMo}_{12}\text{O}_{40}]^{3-}$, $[\text{SiMo}_{12}\text{O}_{40}]^{4-}$, and $[\text{BW}_{12}\text{O}_{40}]^{5-}$ [17, 18], however, metal-dpdo POM compounds using $[\beta\text{-Mo}_8\text{O}_{26}]^{4-}$ as the inorganic component have not been reported so far. As part of our studies on the construction of POM-based inorganic-organic hybrid compounds [19–21], herein we report the synthesis and crystal structure of the coordination polymer $[\text{Cu}(\text{dpdo})_2(\text{H}_2\text{O})_4][\text{Na}_2(\text{Mo}_8\text{O}_{26})\cdot(\text{H}_2\text{O})_6]\cdot(\text{dpdo})\cdot 2\text{H}_2\text{O}$ (**1**) based on 4,4'-bipyridine-*N,N'*-dioxide and $[\beta\text{-Mo}_8\text{O}_{26}]^{4-}$ units.

Experimental Section

General

Materials: All chemicals were of reagent grade quality obtained from commercial sources and used without further purification. 4,4'-Bipyridine-*N,N'*-dioxide (dpdo) was prepared according to the literature method [22].

Instrumentation: Elemental analyses (C, H and N) were carried out on a Perkin-Elmer 240C analytical instrument. IR spectra were recorded on KBr pellets with a Nicolet 170 SXFT-IR spectrophotometer in the 4000–400 cm^{-1} region.

Synthesis

A 5 mL aqueous solution of $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ (1.16 g, 4.8 mmol) and KCl (0.06 g, 0.78 mmol) was adjusted to pH = 6.3 with $12 \text{ mol} \cdot \text{L}^{-1}$ HCl and then heated at 80°C for 10 min and filtered. KCl (1.28 g, 17 mmol) was added. The resulting solution was left for 8 h at room tem-

perature to obtain a colorless solid which was isolated by filtration. A mixture of the colorless solid (0.34 g), $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$ (0.06 g, 0.5 mmol), 4,4'-bipyridine-*N,N'*-dioxide (0.02 g, 0.1 mmol), NaCl (1.46 g, 25 mmol), and 25 mL distilled water was heated at 80°C for 1 h. The resulting mixture was adjusted to pH = 2.8 with HCl ($12 \text{ mol} \cdot \text{L}^{-1}$) and left to evaporate at room temperature to obtain yellow block-shaped crystals suitable for single-crystal X-ray diffraction. The total yield was 30% based on $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$. — Anal. for $\text{C}_{30}\text{H}_{48}\text{CuMo}_8\text{N}_6\text{Na}_2\text{O}_{44}$: calcd. C 17.38, H 2.33, N 4.05; found C 17.42, H 2.35, N 4.02. — IR (cm^{-1} , KBr pellet): $\nu = 3427(\text{s}), 3110(\text{m}), 2924(\text{w}), 2852(\text{w}), 1631(\text{s}), 1550(\text{w}), 1471(\text{s}), 1424(\text{m}), 1385(\text{m}), 1324(\text{w}), 1220(\text{s}), 1181(\text{s}), 1128(\text{w}), 1031(\text{w}), 945(\text{vs}), 915(\text{s}), 839(\text{s}), 714(\text{s}), 661(\text{s}), 555(\text{m}), 516(\text{m}), 479(\text{w}), 449(\text{w}), 414(\text{w})$.

Table 1. Summary of crystal data and refinement results for **1**.

Chemical formula	$\text{C}_{30}\text{H}_{48}\text{CuMo}_8\text{N}_6\text{Na}_2\text{O}_{44}$
Formula weight	2073.78
Temperature, K	296(2)
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	$P\bar{1}$
<i>a</i> , Å	10.586(4)
<i>b</i> , Å	11.535(4)
<i>c</i> , Å	12.447(4)
α , deg	97.410(5)
β , deg	94.445(5)
γ , deg	107.159(3)
Volume, Å ³	1429.4(9)
<i>Z</i>	1
$D_{\text{calcd.}}$, g cm ⁻³	2.41
$F(000)$, e	1009
$\mu(\text{MoK}\alpha)$, mm ⁻¹	2.2
θ range for data collection, deg	1.7–25.00
Index ranges <i>hkl</i>	$-12 \leq h \leq 12, -11 \leq k \leq 13$ $-14 \leq l \leq 12$
Reflections collected	7598
Independent reflections / R_{int}	4992 / 0.172
Data / parameters	4992 / 412
Goodness of fit on F^2	1.075
$R_1(F) / wR_2(F^2) [I > 2\sigma(I)]$	0.0324 / 0.0800
$R_1(F) / wR_2(F^2)$ (all data)	0.0413 / 0.0853
Largest diff. peak / hole, e Å ⁻³	1.13 / -1.77

X-Ray crystallographic study

A suitable crystal of size $0.12 \times 0.15 \times 0.20 \text{ mm}^3$ was chosen for the crystallographic study and mounted on a Bruker Smart APEX II CCD diffractometer. All diffraction measurements were performed at room temperature using graphite-monochromatized $\text{MoK}\alpha$ radiation ($\lambda = 0.71073 \text{ Å}$). The structure was solved by Direct Methods and refined on F^2 by using full-matrix least-squares methods with the program SHELXL/S-97 [23, 24]. All non-hydrogen atoms were refined anisotropically, and all hydrogen atoms were geometrically fixed to allow riding on the parent atoms to which they are attached. Atom O(6W) was refined with restraints for the anisotropic displacement parameters. Space group, lattice parameters and other relevant information are listed in Table 1, and selected bond lengths and angles are given in Table 2.

Bond lengths (Å)			
Na(1)–O(1)	2.451(4)	Na(1)–O(3)	2.418(4)
Na(1)–O(1W)	2.321(6)	Na(1)–O(2W)	2.377(6)
Na(1)–O(3WB)	2.411(5)	Na(1)–O(3W)	2.415(5)
Cu(1)–O(14)	1.931(5)	Cu(1)–O(4W)	2.356(7)
Cu(1)–O(5W)	1.952(5)		
Bond angles (deg)			
O(1W)–Na(1)–O(2W)	85.0(2)	O(2WB)–Na(1)–O(3W)	96.33(18)
O(1WB)–Na(1)–O(3W)	96.1(2)	O(1W)–Na(1)–O(3W)	165.2(2)
O(2W)–Na(1)–O(3W)	107.4(2)	O(3WB)–Na(1)–O(3W)	90.46(16)
O(1W)–Na(1)–O(3)	86.2(2)	O(2W)–Na(1)–O(3)	169.5(2)
O(3WB)–Na(1)–O(3)	78.92(16)	O(3W)–Na(1)–O(3)	82.16(16)
O(1W)–Na(1)–O(1)	85.39(19)	O(2W)–Na(1)–O(1)	104.24(17)
O(3WB)–Na(1)–O(1)	159.43(17)	O(3W)–Na(1)–O(1)	83.85(15)
O(3)–Na(1)–O(1)	80.72(14)	O(14)–Cu(1)–O(5W)	85.7(2)
O(14A)–Cu(1)–O(5W)	94.3(2)	O(14)–Cu(1)–O(4W)	101.0(2)
O(14A)–Cu(1)–O(4W)	79.0(2)	O(5WA)–Cu(1)–O(4W)	92.3(2)
O(5W)–Cu(1)–O(4W)	87.7(2)		

Table 2. Selected bond lengths (Å) and bond angles (deg) for **1**^a.

^a Symmetry code A: $1-x, -y, 1-z$; B: $-x, 1-y, 1-z$.

CCDC 938494 contains the supplementary crystallographic data. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Results and Discussion

IR spectrum

The IR spectrum of polymer **1** exhibits characteristic bands of $[\beta\text{-Mo}_8\text{O}_{26}]^{4-}$. Those at 945, 915, 839 and 714 cm^{-1} should be attributed to stretching vibrations of $\text{Mo}=\text{O}_t$ (terminal oxygen), $\text{Mo}-\text{O}_a(\mu_2)$, $\text{Mo}-\text{O}_b(\mu_3)$ and $\text{Mo}-\text{O}_c(\mu_5)$, which are similar to those of $[\text{Co}(\text{en})_3\text{Mo}_8\text{O}_{26}(\text{H}_3\text{O})\text{Cl}](\text{DMF})_4(\text{H}_2\text{O})$ [25]. The bands at 1471, 1220 and 1181 cm^{-1} are assigned to $\nu(\text{ring})$, $\nu(\text{N}-\text{O})$ and $\delta(\text{C}-\text{H})$ from dpdo, which agrees well with that of related compounds [26]. The band appearing at 3427 cm^{-1} which corresponds to the $\nu(\text{OH})$ stretching frequency indicates the presence of water molecules. These results were finally confirmed by X-ray crystallography.

Crystal and molecular structure

Single-crystal X-ray diffraction analysis has revealed that $[\text{Cu}(\text{dpdo})_2(\text{H}_2\text{O})_4][\text{Na}_2(\text{Mo}_8\text{O}_{26})\cdot(\text{H}_2\text{O})_6]\cdot(\text{dpdo})\cdot 2\text{H}_2\text{O}$ (**1**) is a 1D coordination polymer based on two types of chains. The anionic chain is formed through the coordination interaction between the $[\beta\text{-Mo}_8\text{O}_{26}]^{4-}$ cluster anions and Na^+ cations, and the cationic chain is formed through the coordination interaction of Cu(II) cations and dpdo ligands.

Polymer **1** crystallizes in the triclinic space group $P\bar{1}$, and the asymmetric unit consists of one half $[\beta\text{-Mo}_8\text{O}_{26}]^{4-}$ cluster anion, one Na(I) cation, one Cu(II) cation, one half coordinated dpdo, one uncoordinated dpdo, five coordinated water molecules, and one solvate water molecule. The Cu atom is located on a center of inversion. The coordination environments of the Cu(II) and Na(I) atoms are depicted in Fig. 1. Both have a distorted six-coordinated octahedral geometry.

The polyoxoanion $[\beta\text{-Mo}_8\text{O}_{26}]^{4-}$ is a well-known structure composed of eight distorted edge-sharing MoO_6 octahedra. The Mo–O distances can be

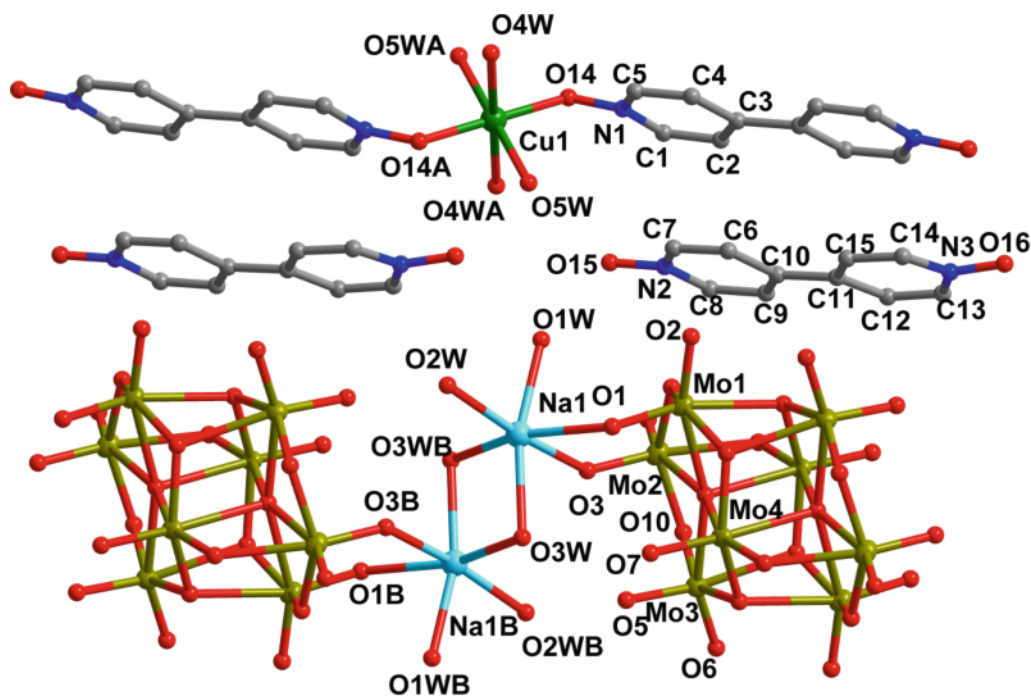


Fig. 1. The structure of **1** and the coordination environment of the Na(I) and Cu(II) centers. Hydrogen atoms and solvate water molecules are omitted for clarity (symmetry codes: A: $1-x, -y, 1-z$; B: $-x, 1-y, 1-z$).

grouped into four sets according to the kind of oxygen atoms bound to the molybdenum atoms, with Mo–O_t (terminal oxygen) of 1.686(3)–1.706(3) Å, Mo–O(μ₂) of 1.746(3)–2.288(3) Å, Mo–O(μ₃) of 1.944(3)–2.364(3) Å, and Mo–O(μ₅) of 2.146(3)–2.479(3) Å. The bond angles O–Mo–O are in the range of 71.44–173.59°.

The Na atom is bound to two oxygen atoms O(1), O(3) of one [β-Mo₈O₂₆]⁴⁻ and four oxygen atoms O(1W), O(2W), O(3W), O(3WA) of four coordinated water molecules. The distances Na–O are in the range of 2.321(6)–2.451(4) Å. Pairs of Na atoms are bridged by a pair of μ-O(3W) donors from two H₂O ligands with an Na(1)···Na(1A) dis-

D–H···A	<i>d</i> (D–H)	<i>d</i> (H···A)	<i>d</i> (D···A)	∠(DHA)	Symmetry codes of atom A
O(1W)–H(1WC)···O(15)	0.85	2.40	3.177(9)	153	–1 + <i>x</i> , <i>y</i> , 1 + <i>z</i>
O(1W)–H(1WD)···O(16)	0.85	1.97	2.692(9)	143	–1 + <i>x</i> , <i>y</i> , <i>z</i>
O(2W)–H(2WC)···O(4W)	0.85	1.97	2.800(11)	165	<i>x</i> , 1 + <i>y</i> , <i>z</i>
O(2W)–H(2WB)···O(5)	0.85	2.04	2.866(6)	164	– <i>x</i> , 1 – <i>y</i> , 1 – <i>z</i>
O(3W)–H(3WC)···O(5)	0.85	2.46	3.057(6)	128	
O(3W)–H(3WC)···O(7)	0.85	2.45	3.230(5)	153	
O(3W)–H(3WD)···O(6W)	0.85	2.07	2.914(8)	176	<i>x</i> , <i>y</i> , 1 + <i>z</i>
O(4W)–H(4WC)···O(6W)	0.85	2.21	3.020(11)	158	<i>x</i> , –1 + <i>y</i> , 1 + <i>z</i>
O(4W)–H(4WA)···O(10)	0.85	2.35	3.107(8)	149	– <i>x</i> , – <i>y</i> , 1 – <i>z</i>
O(5W)–H(5WB)···O(15)	0.85	1.85	2.700(7)	178	<i>x</i> , –1 + <i>y</i> , 1 + <i>z</i>
O(5W)–H(5WA)···O(16)	0.85	1.84	2.663(8)	164	<i>x</i> , –1 + <i>y</i> , <i>z</i>
O(6W)–H(6WC)···O(15)	0.85	1.88	2.695(9)	161	
C(1)–H(1A)···O(1)	0.93	2.38	3.096(8)	134	1 – <i>x</i> , 1 – <i>y</i> , 1 – <i>z</i>
C(2)–H(2A)···O(12)	0.93	2.54	3.445(8)	166	1 – <i>x</i> , 1 – <i>y</i> , 1 – <i>z</i>
C(5)–H(5A)···O(10)	0.93	2.28	2.996(8)	133	– <i>x</i> , – <i>y</i> , 1 – <i>z</i>
C(6)–H(6A)···O(2)	0.93	2.46	3.382(8)	170	1 – <i>x</i> , 2 – <i>y</i> , 1 – <i>z</i>
C(7)–H(7A)···O(6)	0.93	2.37	3.068(7)	132	1 + <i>x</i> , 1 + <i>y</i> , –1 + <i>z</i>
C(8)–H(8A)···O(7)	0.93	2.50	3.250(8)	138	<i>x</i> , <i>y</i> , –1 + <i>z</i>
C(15)–H(15A)···O(2)	0.93	2.38	3.253(8)	157	1 – <i>x</i> , 2 – <i>y</i> , 1 – <i>z</i>

Table 3. Hydrogen bonding interactions (Å and deg) for **1**.

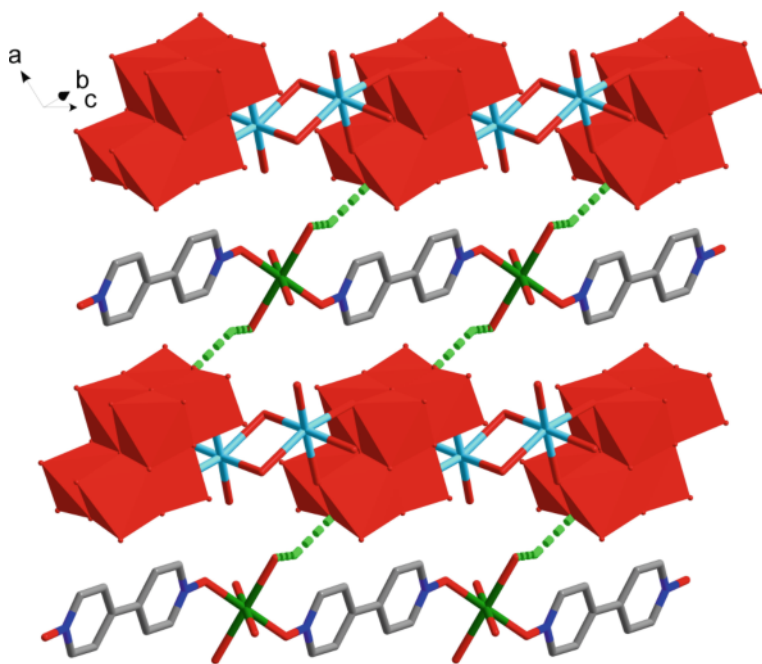


Fig. 2. View of the O(4W)–H(4WA)···O(10) hydrogen bonds (dashed lines) in compound **1**.

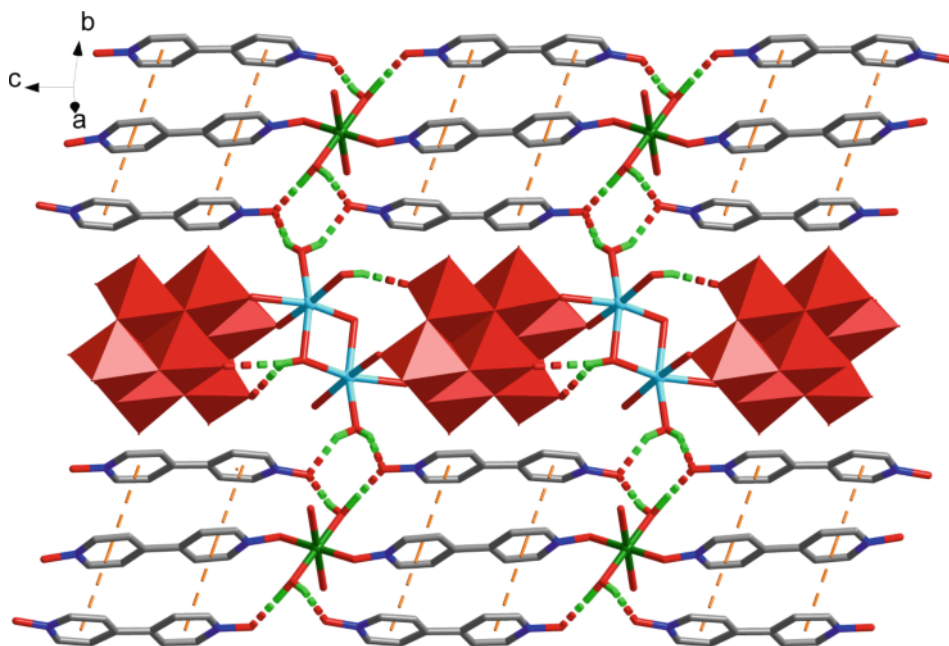


Fig. 3. View of the two types of ionic chain and dpdo molecules in **1** with $\pi \cdots \pi$ interactions and partial O–H \cdots O hydrogen bonds (dashed lines). The solvate water molecules have been omitted.

tance of 3.40 Å and Na(1)–O(3W)–Na(1A) angles of 89.5° (symmetry code: $2-x, 1-y, -1-z$). Consequently, two NaO₆ octahedra are edge-sharing to obtain a dinuclear unit [Na₂]²⁺, which has also been shown previously [27, 28]. Each of these units connects two adjacent [β -Mo₈O₂₆]⁴⁻ anions, and each [β -Mo₈O₂₆]⁴⁻ polyoxoanion acts as a tetradentate ligand to link two [Na₂]²⁺ units to form a one-dimensional anionic structure. The O(2W), O(3W) atoms of water molecules coordinated to Na⁺ cations form three types of O(2W)–H(2WB) \cdots O(5), O(3W)–H(3WC) \cdots O(5) and O(3W)–H(3WC) \cdots O(7) hydrogen bonds with O(5) and O(7) of [β -Mo₈O₂₆]⁴⁻, respectively (Table 3). These hydrogen bonding interactions also play an important role in stabilizing the polymeric structure.

The Cu atom is surrounded by six oxygen atoms from two dpdo ligands [O(14), O(14A)] and four coordinated water molecules [O(4W), O(4WA), O(5W), O(5WA)] to obtain an octahedral coordination geometry. The axial Cu(1)–O(4W) distance is 2.356(7) Å, whereas the average equatorial Cu(1)–O distance is 1.941 Å. These data reveal that the Cu(II) center is subject to a strong Jahn-Teller effect. Adjacent copper cations are linked by bridging dpdo ligands to gener-

ate a 1D cationic structure with Cu \cdots Cu distances of 12.45 Å.

In the solid state, multiform intermolecular hydrogen bonds are formed between the anionic and cationic chains and uncoordinated dpdo components, respectively. Besides the above-mentioned O(2W)–H(2WB) \cdots O(5) hydrogen bonds within the anionic chain, the O(2W) atom as electron donor forms O(2W)–H(2WC) \cdots O(4W) hydrogen bonds with O(4W) of the cationic chain. At the same time, O(4W) is linked to O(10) of [β -Mo₈O₂₆]⁴⁻ of the anionic chain (Fig. 2). The O(15) and O(16) atoms of the uncoordinated dpdo molecule form dual hydrogen bonds with O(1W) of the anionic chain and O(5W) of the cationic chain, respectively. Furthermore, the [N(1)C(1)–C(5)] ring of the coordinated dpdo forms two types of $\pi \cdots \pi$ interactions with the two pyridyl rings [N(2)C(6)–C(10)] and [N(3)C(11)–C(15)] of the uncoordinated dpdos with center-to-center separations of 3.78 and 3.65 Å, dihedral angles of 0.96 and 6.78°, and shortest atom \cdots atom interplanar separations of 3.40 and 3.34 Å. The O(6W) atom of the solvate water molecule forms three types of O–H \cdots O hydrogen bonds with O(3W) of an anionic chain, O(4W) of a cationic chain and O(15) of an uncoordinated

dpdo molecule (Fig. 3). The oxygen atoms of the polyoxoanions feature multiple intermolecular C–H···O hydrogen bonds. Accordingly, these abundant hydrogen bonds and $\pi\cdots\pi$ interactions link adjacent chains and uncoordinated dpdo to form a 3D supramolecular structure.

Conclusions

In summary, we have presented a new example of a metal-dpdo POM compound, $[\text{Cu}(\text{dpdo})_2(\text{H}_2\text{O})_4][\text{Na}_2(\text{Mo}_8\text{O}_{26})\cdot(\text{H}_2\text{O})_6]\cdot(\text{dpdo})\cdot 2\text{H}_2\text{O}$ (**1**), with $[\beta\text{-Mo}_8\text{O}_{26}]^{4-}$ as an inorganic building block. The structure of **1** has been established by single-crystal X-

ray diffraction analysis and also characterized by IR spectroscopy. The coordination polymer consists of anionic and cationic chains. The anionic chain is constructed by the coordination interaction between the $[\beta\text{-Mo}_8\text{O}_{26}]^{4-}$ cluster anions and pairs of Na^+ cations, and the cationic chain is formed through the coordination interaction of Cu^{2+} cations and dpdo ligands.

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