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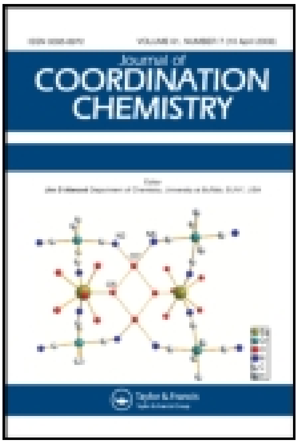

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Two W/Mo complexes containing 2,3-dihydroxynaphthalene: synthesis and structure analysis

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Two W/Mo complexes containing 2,3-dihydroxynaphthalene: synthesis and structure analysis

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Two W/Mo complexes, $[Et_3NH]_2[WO_2(C_{10}H_6O_2)_2]$ (**1**) and $[Et_3NH]_2[MoO_2(C_{10}H_6O_2)_2]$ (**2**), were synthesized by the reaction of $Na_2WO_4 \cdot 2H_2O$ and $(NH_4)_2Mo_2O_7 \cdot 4H_2O$ with 2,3-DHN (DHN = dihydroxynaphthalene) and triethylamine, and characterized by X-ray diffraction and IR. The two complexes exhibit chiral octahedral geometry at central metals, although the overall structure is racemic. Single crystals of **1** are more difficult to obtain than for **2**. The molybdenum complex can be easily isolated at room temperature (around 25°C), while the tungsten one needs to be isolated under 15°C, otherwise, the quality of crystal will be poor, leading to disorder. The structures of these two complexes are chiral and enantiomorphous to each other.

Keywords: Tungsten complex; Molybdenum complex; 2,3-Dihydroxynaphthalene; Triethylamine; Chiral; Enantiomorphous

1. Introduction

Tungsten and molybdenum have potential applications in ion exchange, optical, electro, magnetic materials, and medicine [1–7]. In biocatalysis systems, their corresponding transfer-enzymes play vital roles in global cycles of nitrate, carbon, and sulfur [8, 9]. X-ray analysis has shown that the active structure factors of the corresponding transfer-enzymes are coordinated by the metal (W/Mo) and the sulfur of dithiolene [10, 11]. Monomeric complexes containing oxo-ligands exhibit diverse compositions and significant structural versatility [12–17]. Herein, we synthesized two complexes, in which W/Mo coordinated with 2,3-DHN forming the core structures, possibly simulating the corresponding tungstoenzyme cofactor and molybdoenzyme cofactor. The two crystal structures have a chiral enantiomorphous character, a phenomenon seldom reported.

Our laboratory has synthesized such complexes and published three crystal structures [18–20]. However, in some, there are small organic molecules like six-member ring cations $(C_5H_{11}N_2)^+$ forming, which, we think, might affect the biological activity of

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these complexes, so we tried triethylamine in order to avoid the formation of such small organic molecules. First we obtained **1**, and then we made a parallel experiment using Mo to replace W in **1**, forming $[MoO_2(C_{10}H_6O_2)_2]^{2-}$, **2**. Both exhibit chiral octahedral geometry at central metals being structurally enantiomorphous. Single crystals of **1** are harder to obtain than for **2**.

2. Experimental

All reagents and materials used for synthesis were of reagent grade and used without purification. IR spectra were recorded with KBr pellets on a Bruker EQUINOX 55 FT-spectrophotometer.

2.1. *Synthesis of* $[Et_3NH]_2[WO_2(C_{10}H_6O_2)_2]$ *(1)*

Single crystals of **1** were synthesized from a mixture of sodium tungstate, 2,3-DHN, triethylamine, methyl alcohol, and acetonitrile. A typical synthesis was as follows: 0.1 g 2,3-DHN was dissolved in 15 mL CH_3OH, and then 15 mL CH_3CN, 0.2 g $Na_2WO_4 \cdot 2H_2O$, and 8 mL triethylamine were successively added at 15°C with stirring; after 24 h, the solution was filtered and the yellow filtrate was layered with $(CH_3)_2CHOH$. Yellow crystals were obtained in 2 weeks. IR (cm^{-1}, KBr): 3431, 3016, 2985, 1574, 1475, 1393, 1333, 1270, 1162, 1073, 904, 836, 741, 645, 623, 557, 479.

2.2. *Synthesis of* $[Et_3NH]_2[MoO_2(C_{10}H_6O_2)_2]$ *(2)*

Single crystals of **2** were synthesized from a mixture of ammonium molybdate, 2,3-DHN, triethylamine, methyl alcohol, and acetonitrile. A typical synthesis was as follows: 0.35 g 2,3-DHN was dissolved in 30 mL CH_3OH, and then 30 mL CH_3CN, 0.45 g $(NH_4)_2Mo_2O_7 \cdot 4H_2O$, and 8 mL triethylamine were successively added at room temperature (around 25°C) with stirring; after 8 h, the solution was filtered and the dark-red filtrate was layered with Et_2O. Dark-red crystals were obtained in one week. IR (cm^{-1}, KBr): 3453, 3019, 2703, 1582, 1484, 1327, 1313, 1262, 1103, 1062, 930, 905, 813, 745, 672, 644, 630, 605, 498.

2.3. *X-ray crystallography*

Single-crystal structure determination by X-ray diffraction was performed on a Bruker Apex II CCD area detector device with Mo-Kα monochromated radiation ($\lambda = 0.71073$ Å) at room temperature. The structure was solved by direct methods with SHELXS-97 and Olex 2 [21, 22]. An absorption correction based on symmetry equivalent reflections was applied by using SADABS [23]. The final refinement included atomic positions for all atoms and anisotropic thermal parameters for all non-hydrogen atoms. Full-matrix least-squares structure refinement against $|F^2|$ was carried out with the SHELXTL-PLUS package of programs. Crystallographic data for **1** and **2** are summarized in table 1.

Table 1. Crystal data summaries of intensity data collection and structure refinement for **1** and **2**.

	1	**2**
Empirical formula	$C_{32}H_{44}N_2O_6W$	$C_{32}H_{44}N_2O_6Mo$
Formula weight	736.54	648.63
Crystal system	Orthorhombic	Orthorhombic
Crystal color, habit	Yellow, block	Dark-red, block
Temperature (K)	298.2	296.0
Space group	*Pbcn*	*Pbcn*
Unit cell dimensions (Å, °)		
a	25.494(2)	25.390(3)
b	9.8782(7)	9.9012(10)
c	12.9496(11)	12.9484(13)
α	90.00	90.00
β	90.00	90.00
γ	90.00	90.00
Volume (Å^3), Z	3261.2(5), 4	3255.1(6), 4
Calculated density, ρ_{calc} (Mg m^{-3})	1.500	1.324
Absorption coefficient, μ (mm^{-1})	3.585	0.446
$F(000)$	1488	1360
Crystal size (mm^3)	$0.32 \times 0.26 \times 0.18$	$0.3 \times 0.3 \times 0.2$
Theta range (°)	2.71–25.02	1.60–25.02
Index ranges	$-22 \le h \le 30$; $-11 \le k \le 11$; $-14 \le l \le 15$	$-30 \le h \le 26$; $-11 \le k \le 8$; $-15 \le l \le 8$
Reflections collected	15,904	12,675
Independent reflections	2878[R(int) = 0.0464]	2876[R(int) = 0.0254]
Data/restraints/parameters	2878/0/189	2876/91/189
Goodness-of-fit on F^2	1.102	1.047
Final R indexes [$I > 2\sigma(I)$]	$R_1 = 0.0303$ $wR_2 = 0.0602$	$R_1 = 0.0271$ $wR_2 = 0.0747$
Final R indexes [all data]	$R_1 = 0.0647$ $wR_2 = 0.0801$	$R_1 = 0.0366$ $wR_2 = 0.0827$
Largest difference peak/hole (e Å^{-3})	0.992/−0.772	0.208/−0.313

Values in parentheses for reflections with $I > 2\sigma$ (I). $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)]\}^{1/2}$ and $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP)]$, where $P = (2F_c^2 + F_o^2)/3$.

Table 2. Characteristic IR data (cm^{-1}) of **1** and **2**.

	M = O stretches	O–C stretch	C–H stretch	C–H bend
Complex **1**	836	1270	3016	741
Complex **2**	905	1262	3019	745

M = W, Mo.

3. Results and discussion

3.1. *Synthesis*

Parallel experiments show that the molybdenum complex can be easily obtained at room temperature (around 25°C), while **1** needs to be synthesized and isolated at lower temperature. At the same temperature (around 25°C), the structure of **1** always shows disorder. In order to obtain crystals with better quality, we adjusted the temperature to 15°C to reduce the growth rate of **1** in the whole process. Because crystals easily form defects when the growth rate of crystals are faster than that of crystal nuclei, we

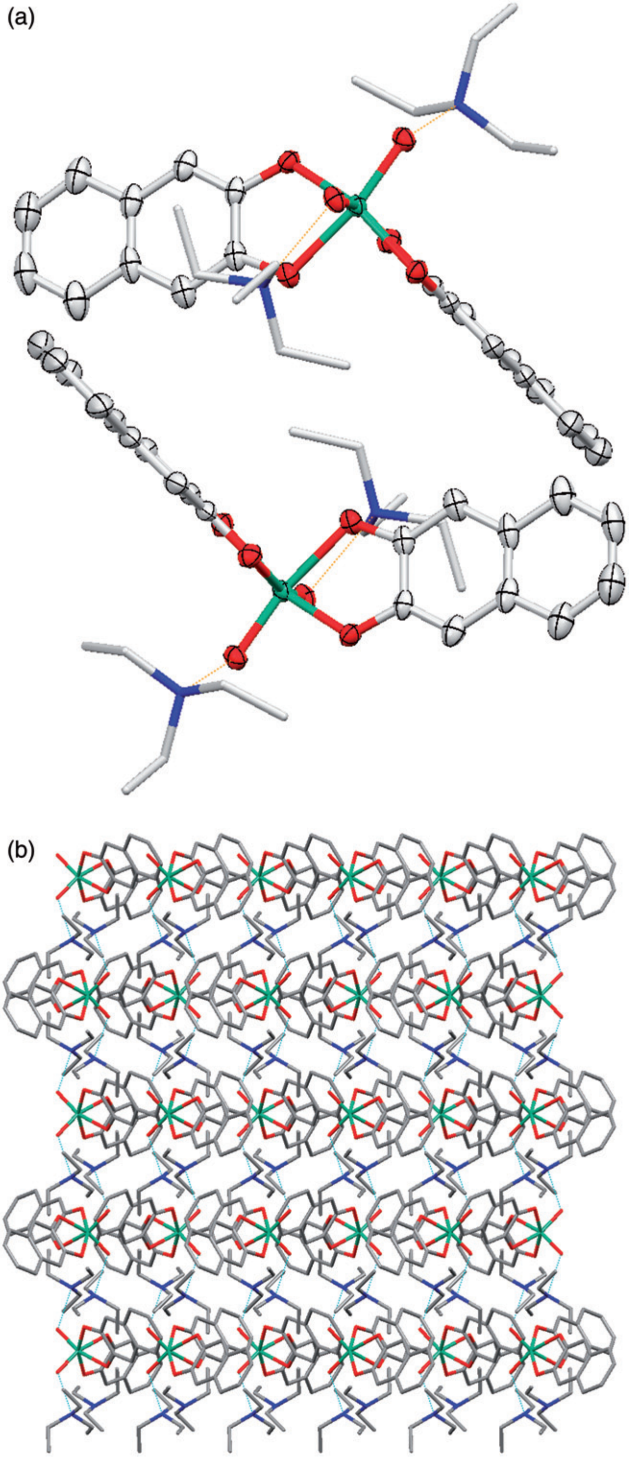

Figure 1. (a) The packing block of **1** and **2**: each chiral $[MoO_2(C_{10}H_6O_2)_2]^{2-}$ coupled to its enantiomer. (b) Packing diagram along the *a*-axis. (c) Packing diagram along the *c*-axis. (d) Packing diagram along the *b*-axis.

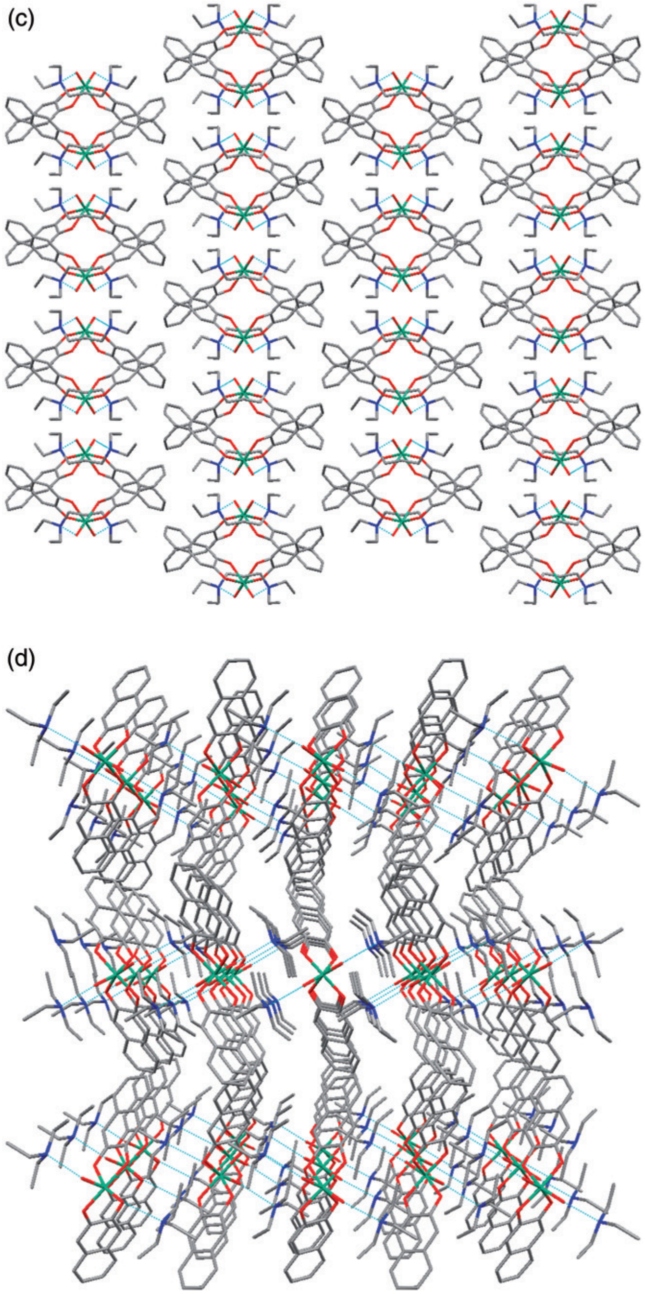

Figure 1. Continued.

conjecture that the binding power of W to oxo is weaker than that of Mo. Summarily, we conclude that the single crystal of **1** was much harder to obtain than that of **2**.

Table 3. Bond lengths (Å) and angles (°) for **1** and **2**.

Complex **2**[b]			
Bond lengths			
W1–O1	1.984(4)	C2–C3	1.420(10)
W1–O1^{1}	1.984(4)	C3–C4	1.375(9)
W1–O2	2.105(4)	C4–C5	1.425(10)
W1–O2^{1}	2.105(4)	C5–C6	1.405(11)
W1–O3	1.760(4)	C5–C10	1.421(10)
W1–O3^{1}	1.760(4)	C6–C7	1.438(10)
N1–C11	1.499(9)	C7–C8	1.361(13)
N1–C13	1.482(9)	C8–C9	1.345(13)
N1–C15	1.494(10)	C9–C10	1.393(12)
O1–C2	1.349(8)	C11–C12	1.452(13)
O2–C3	1.345(8)	C13–C14	1.506(10)
C1–C2	1.347(9)	C15–C16	1.457(12)
C1–C6	1.406(11)		
Bond angles			
N1–C13–C14	114.4(7)	C1–C6–C7	121.7(9)
O1–W1–O1^{1}	160.5(3)	C2–O1–W1	120.0(4)
O1^{1}–W1–O2	88.97(18)	C2–C1–C6	120.3(8)
O1–W1–O2^{1}	88.97(18)	C3–O2–W1	115.1(4)
O1^{1}–W1–O2^{1}	76.26(18)	C3–C4–C5	119.1(8)
O1–W1–O2	76.26(18)	C4–C3–C2	119.7(7)
O1–C2–C3	113.6(6)	C5–C6–C1	119.3(8)
O2–W1–O2^{1}	82.3(2)	C5–C6–C7	119.0(8)
O2–C3–C2	114.9(6)	C6–C5–C4	120.0(7)
O2–C3–C4	125.3(8)	C6–C5–C10	119.1(9)
O3^{1}–W1–O1^{1}	103.96(19)	C8–C7–C6	119.1(10)
O3^{1}–W1–O1	88.4(2)	C8–C9–C10	120.7(10)
O3–W1–O1	103.96(19)	C9–C8–C7	122.7(10)
O3–W1–O1^{1}	88.4(2)	C9–C10–C5	119.5(10)
O3–W1–O2	89.48(19)	C10–C5–C4	120.9(9)
O3^{1}–W1–O2^{1}	89.48(19)	C12–C11–N1	113.0(8)
O3^{1}–W1–O2	162.64(19)	C13–N1–C11	113.0(6)
O3–W1–O2^{1}	162.64(19)	C13–N1–C15	114.9(7)
O3–W1–O3^{1}	102.2(3)	C15–N1–C11	107.7(7)
C1–C2–O1	124.9(7)	C16–C15–N1	114. 5(8)
C1–C2–C3	121.5(7)		

Complex **2**[b]			
Bond lengths	1.9987(15)	C2–C3	1.369(3)
Mo1–O1^{1}	1.9988(15)	C3–C4	1.428(4)
Mo1–O1	2.1127(14)	C4–C5	1.416(4)
Mo1–O2^{1}	2.1127(14)	C4–C7	1.422(4)
Mo1–O2	1.7270(14)	C5–C6	1.410(4)
Mo1–O3^{1}	1.7270(14)	C5–C10	1.411(4)
Mo1–O3	1.346(3)	C7–C8	1.383(5)
O1–C1	1.329(3)	C8–C9	1.371(5)
O2–C2	1.488(3)	C9–C10	1.351(5)
N1–C11	1.489(3)	C11–C12	1.476(5)
N1–C13	1.499(3)	C13–C14	1.513(4)
N1–C15	1.426(3)	C15–C16′	1.463(5)
C1–C2	1.365(3)		
C1–C6			
Bond angles			
O1^{1}–Mo1–O1	160.69(9)	C1–C6–C5	120.2(2)
O1^{1}–Mo1–O2^{1}	76.03(6)	C2–O2–Mo1	115.82(13)

(*Continued*)

Table 3. Continued.

Complex **2**[b]			
O1–Mo1–O2	76.03(6)	C2–C3–C4	120.2(3)
O1^1–Mo1–O2	89.48(6)	C3–C2–C1	119.1(2)
O1–Mo1–O2	89.48(6)	C5–C4–C3	119.9(2)
O1–C1–C2	114.43(18)	C5–C4–C7	119.0(3)
O1–C1–C6	123.8(2)	C6–C1–C2	121.7(2)
O2–Mo1–O2^1	83.33(8)	C6–C5–C4	118.9(2)
O2–C2–C1	114.47(19)	C6–C5–C10	122.9(3)
O2–C2–C3	126.4(2)	C7–C4–C3	121.1(3)
O3^1–Mo1–O1^1	105.23(7)	C8–C7–C4	119.5(3)
O3^1–Mo1–O1	86.98(7)	C9–C8–C7	121.0(3)
O3–Mo1–O1	105.23(7)	C9–C10–C5	121.6(4)
O3–Mo1–O1^1	86.98(7)	C10–C5–C4	118.2(3)
O3^1–Mo1–O2	161.48(6)	C10–C9–C8	120.7(3)
O3–Mo1–O2^1	161.48(6)	C11–N1–C13	113.2(2)
O3^1–Mo1–O2^1	89.29(6)	C11–N1–C15	108.6(2)
O3–Mo1–O2	89.29(6)	C12–C11–N1	114.2(3)
O3–Mo1–O3^1	102.44(10)	C13–N1–C15	115.1(2)
N1–C13–C14	113.5(2)	C16–C15–N1	114.8(3)
C1–O1–Mo1	119.20(13)		

Symmetry transformations used to generate equivalent atoms: [a]^{1}1 − *X*, +*Y*, 3/2 − *Z*; [b]^1−*X*, +*Y*, 3/2 − *Z*.

Table 4. H-bond parameters of **1** and **2**.

	Donor–H ··· acceptor	D–H (Å)	H ··· A (Å)	D ··· A (Å)	D–H ··· A (°)
Complex **1**	N(1)–H(1) ··· O(3)	0.909	1.832	2.732	169.751
Complex **2**	N(1)–H(1) ··· O(3)	0.910	1.914	2.803	164.942

3.2. *IR spectra of complexes*

IR spectra of the complexes showed two strong peaks attributable to the asymmetric and symmetric metal–oxo stretches in a *cis*-moiety. Other important frequency bands are attributable to O–C stretch, C–H stretch, and C–H bend. All the IR stretches are listed in table 2.

3.3. *Crystal structure*

Complexes **1** and **2** are both orthorhombic in *Pbcn* space group. The perspective views of them and their packing diagrams are shown in figure 1, and their bond lengths and angles are given in table 3.

From the molecular structures, **1** and **2** are *cis*-dioxo-monomeric complexes with the ratio of metal to ligand 1 : 2. Both central metals are in a distorted octahedral coordination environment with two oxo's forming terminal M–O (M = W, Mo) bonds and four oxygens of two naphthalene-2,3-diols, giving coordination number six.

The length of W = O is 1.832 Å, and two [Et$_3$NH]$^+$ connect with [WO$_2$(C$_{10}$H$_6$O$_2$)$_2$]$^{2-}$ *via* N(1)–H(1) ··· O(3) (2.732 Å, 169.751°), and the lengths of the four W–O (2,3-DHN)

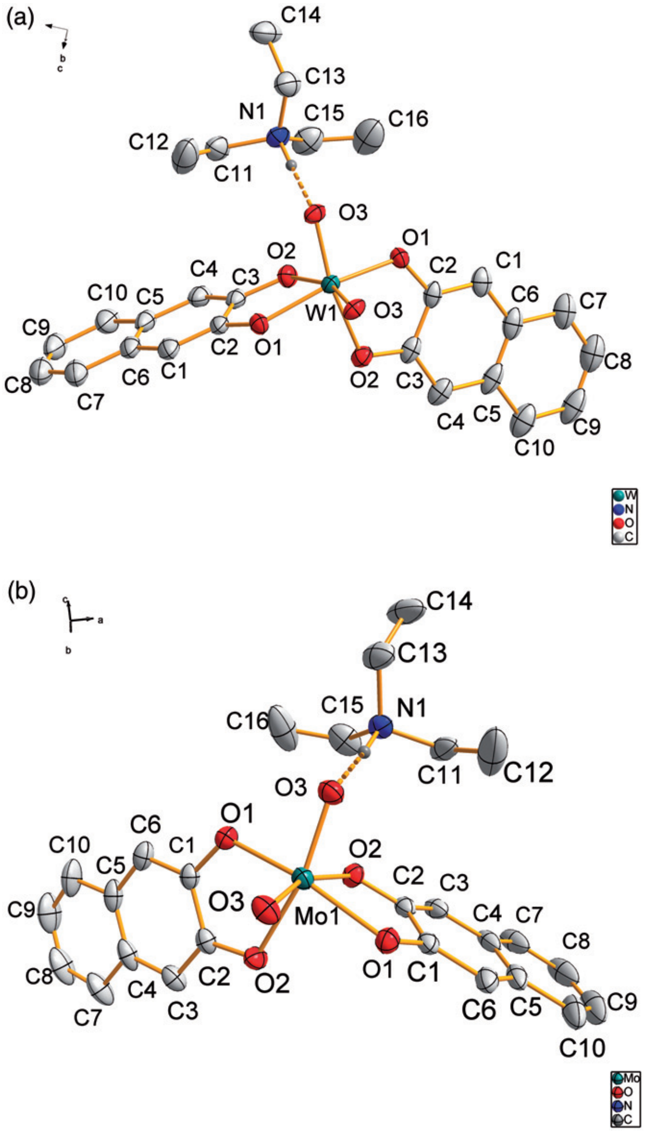

Figure 2. Molecular structures of **1** and **2** with thermal ellipsoids drawn at 30% probability level: (a) corresponds to **1**: Λ-configuration; (b) corresponds to **2**: Δ-configuration; they structurally resemble two chiral enantiomorphous parts.

bonds are 1.984, 2.105, 1.984, and 2.105 Å; angles around W are in the range 76.273–162.655° (table 3).

The length of Mo = O is 1.727 Å, and two $[Et_3NH]^+$ connect with $[MoO_2(C_{10}H_6O_2)_2]^{2-}$ *via* N(1)–H(1) ··· O(3) (2.803 Å, 164.942°), and the lengths of the four Mo–O (2,3-DHN) bonds are 1.999, 2.113, 1.999, and 2.113 Å with angles around Mo in the range 76.036–160.699° (table 3). The structure was stabilized by hydrogen bonds (table 4) and van der Waals forces.

The two complexes structurally show chiral and enantiomorphous characteristics. As shown in figure 2(a), $[WO_2(C_{10}H_6O_2)_2]^{2-}$ adopts Λ-configuration, while in figure 2(b), $[MoO_2(C_{10}H_6O_2)_2]^{2-}$ exhibits Δ-configuration [24, 25].

In addition, each chiral $[MoO_2(C_{10}H_6O_2)_2]^{2-}$ is coupled to its enantiomer in the space giving a racemic structure (figure 1a). As shown in figure 1(b), the packing motifs further constitute a monolayer-chain along the *a*-axis, in which two neighboring monolayer-chains array anti-parallel, exhibiting a 2-D rhombus-grid-like network along the *c*-axis (figure 1c). Viewing along the *b*-axis, the pairs of enantiomers are packed through van der Waals forces, forming a vertical and open laminated structure in the crystal lattice (figure 1d).

4. Conclusion

We synthesized two W/Mo complexes, $[Et_3NH]_2[WO_2(C_{10}H_6O_2)_2]$ (**1**) and $[Et_3NH]_2[MoO_2(C_{10}H_6O_2)_2]$ (**2**), and characterized them by X-ray and IR. From a lot of parallel experiments, we find that single crystals of **1** are much harder to obtain than the corresponding molybdenum crystals, probably arising from the coordination ability of metal salts. Even though the structures of **1** and **2** are almost the same, coincidentally, they structurally resemble two chiral enantiomorphous parts: $[WO_2(C_{10}H_6O_2)_2]^{2-}$ adopts Λ-configuration, while $[MoO_2(C_{10}H_6O_2)_2]^{2-}$ exhibits Δ-configuration. Each chiral $[MoO_2(C_{10}H_6O_2)_2]^{2-}$ anion is coupled to its enantiomer, and they arrange as anti-parallel layers and integrally assemble into a 2-D rhombus-grid-like network in the space.

Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center (CCDC), No. 780673 for **1** and No. 780617 for **2**. Copies of the data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

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