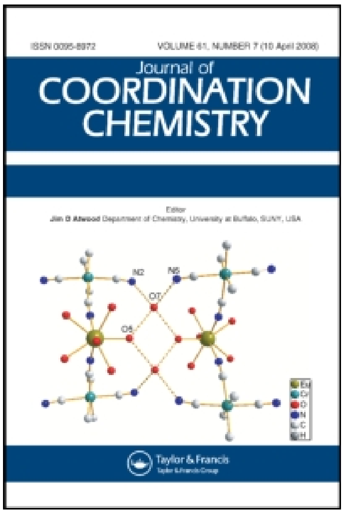

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Synthesis and structures of <i>cis</i> -WO$_2$ complexes with 2,3-dihydroxynaphthalene

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Synthesis and structures of *cis*-WO_2 complexes with 2,3-dihydroxynaphthalene

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$\{Na(OCH_3)[H_3N(CH_2)_2NH_2]_2\}[WO_2(C_{10}H_6O_2)_2]$ (**1**) was obtained by the reaction of $Na_2WO_4 \cdot 2H_2O$ with 2,3-dihydroxynaphthalene and ethylenediamine. $[H_2N(CH_2)_3NH_3]_2[WO_2(C_{10}H_6O_2)_2]$ (**2**) was synthesized by the reaction of $Na_2WO_4 \cdot 2H_2O$ with 2,3-dihydroxynaphthalene and 1,3-propylenediamine. Complex **1** was a one-dimensional chain-like structure and the Na atom is in the structure, while complex **2** was a discrete monomer without Na in its structure. The two complexes were synthesized in the same reaction conditions, except that protonated ethylenediamine was used in reaction 1, but 1,3-propylenediamine in reaction 2.

Keywords: *Cis*-dioxotungsten complexes; 2,3-Dihydroxynaphthalene; Ethylenediamine; 1,3-Propylenediamine; Crystal structure

1. Introduction

The chemistry of transition metal-oxo compounds is an area of permanent interest that has experienced a renaissance [1–4]. Numerous monomeric complexes containing terminal oxo-ligands have been prepared and investigated due to their potential applications as catalysts in industry epoxidation reactions and other oxo transfer processes [5–8]. Tungsten is a versatile transition element possessing a large number of stable and accessible oxidation states. High-valent oxo-tungsten complexes have received attention for their use in various catalytic processes such as alcohol and olefin oxidation, olefin–epoxidation and metathesis reactions [9–15]. Recent findings concerning the biological roles of tungsten have heightened interest in this class of compounds, in particular those containing dithiolene-type ligands [16–18]. Such compounds with dithiolene-type ligands are related to a class of enzymes known as oxotransferases, which catalyze oxygen atom transfer to and from a substrate. It is generally believed that most of these organic transformations

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and biological processes involve oxygen atom transfer reactions. Oxo- and dioxotungsten complexes that are able to undergo oxo-transfer reactions are thus of special importance. However, in contrast to the related molybdenum chemistry, tungsten-mediated atom transfer reactions have been little studied, even though in all of these industrial and biological reactions, tungsten plays a role in the catalytic redox site.

We reported the syntheses and crystal structures of *cis*-dioxo-tungsten(VI) complexes, from interaction of sodium tungstate, catechol, ethylenediamine (1,2-propylenediamine or 1,3-propylenediamine). A homologue of catechol–2,3-dihydroxynaphthalene has rarely been reported with *cis*-dioxo-tungsten(VI). Our laboratory synthesized such complexes and published the synthesis and crystal structure of one [19]. As a sequel, we recently synthesized two dioxotungsten complexes: $\{Na(CH_3O)[H_3N(CH_2)_2NH_2]_2\}[WO_2(C_{10}H_6O_2)_2]$ (**1**) and $[H_2N(CH_2)_3NH_3]_2[WO_2(C_{10}H_6O_2)_2]$ (**2**).

2. Experimental

All reagents and materials used for synthesis were reagent grade and used without further purification. IR spectra were recorded with KBr pellets on a Bruker EQUINOX 55 FT-spectrophotometer. UV-vis spectra were recorded in a 10 mm quartz cell on a Shimadzu UV-265 instrument.

2.1. *Synthesis of (1)*

Single crystals of **1** were synthesized from a mixture of sodium tungstate, 2,3-dihydroxynaphthalene, ethylenediamine, methyl alcohol and acetonitrile. A typical synthesis was as follows: 0.3 g 2,3-dihydroxynaphthalene was dissolved in 15 mL CH_3OH, and then 15 mL CH_3CN, 0.3 g $Na_2WO_4 \cdot 2H_2O$ and 8 mL ethylenediamine were successively added at room temperature with stirring; 20 h later, the solution was filtered and the yellow filtrate was layered with Et_2O. Light orange needle-like crystals were obtained in several weeks. IR (cm^{-1}, KBr): 3360, 3184, 2931, 1578, 1467, 1260, 1156, 956, 917, 868, 754, 647, 623, 558, 481.

2.2. *Synthesis of (2)*

Single crystals of **2** were synthesized from a mixture of sodium tungstate, 2,3-dihydroxynaphthalene, 1,3-propylenediamine, methyl alcohol and acetonitrile. A typical synthesis was as follows: 0.3 g 2,3-dihydroxynaphthalene was dissolved in 15 mL CH_3OH, and then 15 mL CH_3CN, 0.3 g $Na_2WO_4 \cdot 2H_2O$ and 6 mL 1,3-propylenediamine were successively added at room temperature with stirring; 20 h later, the final solution was filtered and the yellow filtrate was layered with Et_2O. Light orange crystals were obtained in several weeks. IR (cm^{-1}, KBr): 3414, 3049, 2927, 1576, 1466, 1261, 1169, 1021, 914, 867, 754, 646, 623, 556, 480.

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 radiation. The structure was solved by direct methods with SHELXS-97. An absorption correction based on symmetry equivalent reflections was applied by using the SADABS program. The final refinement included atomic positions for all the atoms and anisotropic thermal parameters for all the 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.

3. Results and discussion

3.1. *IR spectra of complexes*

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

Table 1. Summary of crystallographic data and parameters for **1** and **2**.

	1	**2**
Molecular formula	$C_{26}H_{31}N_4NaO_8W$	$C_{26}H_{36}N_4O_6W$
Formula weight	734.38	684.43
Temperature (K)	273(2)	273(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Orthorhombic	Monoclinic
Space group	*Pbca*	*P*2(1)/*c*
Unit cell dimensions (Å, °)		
a	14.5679(3)	7.6533(2)
b	15.3703(3)	32.4761(7)
c	27.2360(5)	12.0074(2)
α	90	90
β	90	103.7820(10)
γ	90	90
Volume(Å^3)	6098.5(2)	2898.51(11)
Z	8	4
Calculated density (Mg m^{-3})	1.6	1.568
Absorption coefficient (mm^{-1})	3.853	4.029
F(000)	2912	1368
Crystal size (mm^3)	0.30 × 0.30 × 0.20	0.14 × 0.13 × 0.12
θ range for data collection (°)	2.05–28.33	1.25–28.30
Index ranges	$-19 \leq h \leq 19$, $-20 \leq k \leq 20$, $-36 \leq l \leq 36$	$-10 \leq h \leq 10$, $-43 \leq k \leq 43$, $-16 \leq l \leq 16$
Reflections collected/unique	92258/7554 [$R_{(int)} = 0.0901$]	27936/7190[$R_{(int)} = 0.0425$]
Completeness	99.4% ($\theta = 28.33°$)	99.7% (θ=28.30°)
Data/restraints/parameters	7554/0/364	7190/0/338
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0408$, $\omega R_2 = 0.1238$	$R_1 = 0.0394$, $\omega R_2 = 0.1310$
R indices (all data)	$R_1 = 0.0930$, $\omega R_2 = 0.1502$	$R_1 = 0.0587$, $\omega R_2 = 0.1434$
GOF on F^2	0.844	0.986
Largest diff. peak and hole (e · Å^{-3})	0.912 and −1.779	2.005 and −0.957

Values in parentheses for reflections with $I > 2.0\sigma(I)$. $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o.|$ $wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$ and $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP)]$, where $P = (2F_c^2 + F_o^2)/3$.

Table 2. Characteristic vibrations for **1** and **2**.

	W=O stretches	O–C stretch	C–H stretch	C–H bend
Complex **1**	868–917	1156	3184	754
Complex **2**	867–914	1169	3049	754

3.2. *Crystal structure of (1)*

Selected bond lengths and angles of **1** are listed in table 3, and its molecular structure and packing diagram are shown in figure 1(a) and (b), respectively.

Complex **1** crystallizes in the orthorhombic system with Pbca space group. Tungsten is in a distorted octahedral coordination environment, common to nearly all structurally characterized six-coordinate W(VI)O_2 species [9, 20–22]. The tungsten center coordinates two O atoms as two terminal W–O {W(1)–O(3) and W(1)–O(4)} with an O(3)–W(1)–O(4) angle of 103.5(2) and four oxygen donor atoms from chelating 2,3-dihydroxynaphthalene with W–O bond distances in the range 1.989(4)–2.154(4) Å. The two 2,3-dihydroxynaphthalene molecules are in two different planes and the dihedral angle between them is 82.9°.

The Na atom bonds with W through a terminal O atom with W(1)–O(3)–Na(1) bond angle of 167.8(3)° and a shorter W–O bond distance of 1.729(5) Å. The Na atom also bonds with N atoms from two protonated ethylenediamines and an O atom from methanol. The Na–N bond distances were within the range 2.494(7)–2.665(8) Å and Na(1)–O(7) bond distance was 2.349(8) Å.

The chain-like structure was assembled by–W–O–Na–O along the c axis. The O_2–W_1–O_3 bond angle was 164.7(2)°. Two molecular chains were aligned anti-parallel. Each molecular chain has many $[WO_2(C_{10}H_6O_2)_2]^{2-}$ units linked through π-π stacking interactions along the c axis; the 1D chain-like structures are building blocks assembled into a double-lamellar architecture in the crystal lattice. Chain-like molecules might assemble as nano-threads, nano-belts and nano-lamellar architectures through hydrogen bonds, π–π stacking interaction and Van der Waals forces.

3.3. *Crystal structure of (2)*

Selected bond lengths and angles of **2** are listed in table 4, and its molecular structure and packing diagram exhibited in figure 2(a–c).

The geometry around tungsten is a distorted octahedron coordinated by two O atoms forming terminal W–O bonds and four O atoms of two 2,3-bihydroxynaphthalenes. The terminal metal-oxo distances were within the range 1.738(4) Å–1.749(5) Å, and the four W–O bond distances were not equivalent, divided into two sorts: W–O(1),W–O(6) with longer bond length [2.138(4)] Å, *trans* to the terminal O atoms and W–O(2), W–O(5) with shorter bond length [1.979(4)] Å, *cis* to the terminal O atoms. The O(3)–W–O(4) angle of 101.8(2)° enhanced π interactions between the oxo and metal orbital [23]. There were two protonated 1,3-propylenediamines to compensate the anion $[WO_2(OC_{10}H_6O)_2]^{2-}$.

As shown in figure 2(a), the metal center coordinates to 2,3-dihydroxynaphthalene ligands, with $[W(VI)O_2(C_{10}H_6O_2)_2]^{2-}$ possessing chiral character (Λ or Δ) aligned

Table 3. Selected bond lengths (Å) and angles (°) for **1**.

W(1)–O(3)	1.729(5)	O(3)–Na(1)	2.392(5)
W(1)–O(4)	1.746(4)	Na(1)–O(7)	2.349(8)
W(1)–O(1)	1.989(4)	Na(1)–O(2)#2	2.414(5)
W(1)–O(5)	2.002(4)	Na(1)–N(1)	2.494(7)
W(1)–O(6)	2.110(4)	Na(1)–N(4)	2.665(8)
W(1)–O(2)	2.154(4)	Na(1)–O(4)#2	2.673(6)
W(1)–Na(1)#1	3.589(3)	Na(1)–W(1)#2	3.589(3)
C(1)–O(6)	1.324(7)	N(2)–C(28)	1.463(8)
C(9)–O(2)	1.332(7)	N(1)–C(29)	1.432(1)
C(8)–O(1)	1.364(7)	O(7)–C(32)	1.345(1)
C(2)–O(5)	1.368(7)	N(4)–C(30)	1.433(1)
O(4)–Na(1)#1	2.673(6)	C(31)–N(5)	1.484(9)
O(2)–Na(1)#1	2.414(5)	C(35)–O(9)	1.895(2)
O(3)–W(1)–O(4)	103.5(2)	O(6)–W(1)–O(2)	81.1(2)
O(3)–W(1)–O(1)	89.1(2)	O(3)–W(1)–Na(1)#1	149.0(2)
O(4)–W(1)–O(1)	108.1(2)	O(4)–W(1)–Na(1)#1	45.5(2)
O(3)–W(1)–O(5)	101.6(2)	O(1)–W(1)–Na(1)#1	99.7(1)
O(4)–W(1)–O(5)	88.3(2)	O(5)–W(1)–Na(1)#1	81.1(1)
O(1)–W(1)–O(5)	158.0(2)	O(6)–W(1)–Na(1)#1	116.3(1)
O(3)–W(1)–O(6)	94.0(2)	O(2)–W(1)–Na(1)#1	40.9(1)
O(4)–W(1)–O(6)	158.3(2)	O(6)–C(1)–C(6)	126.7(6)
O(1)–W(1)–O(6)	84.7(2)	O(6)–C(1)–C(2)	114.4(6)
O(5)–W(1)–O(6)	75.5(2)	O(2)–C(9)–C(10)	126.3(6)
O(3)–W(1)–O(2)	164.7(2)	O(2)–C(9)–C(8)	115.1(5)
O(4)–W(1)–O(2)	85.0(2)	C(13)–C(8)–O(1)	123.5(5)
O(1)–W(1)–O(2)	76.1(2)	O(1)–C(8)–C(9)	114.1(5)
O(5)–W(1)–O(2)	91.2(2)	O(5)–C(2)–C(1)	113.5(6)
C(2)–O(5)–W(1)	119.4(4)	O(7)–Na(1)–O(4)#2	76.8(2)
C(1)–O(6)–W(1)	116.8(4)	O(3)–Na(1)–O(4)#2	166.3(2)
W(1)–O(4)–Na(1)#1	106.7(2)	O(2)#2–Na(1)–O(4)#2	62.6(1)
C(8)–O(1)–W(1)	119.8(4)	N(1)–Na(1)–O(4)#2	90.5(2)
C(9)–O(2)–W(1)	114.7(3)	N(4)–Na(1)–O(4)#2	95.5(2)
C(9)–O(2)–Na(1)#1	113.2(4)	O(7)–Na(1)–W(1)#2	76.4(2)
W(1)–O(2)–Na(1)#1	103.4(2)	O(3)–Na(1)–W(1)#2	138.7(2)
W(1)–O(3)–Na(1)	167.8(3)	O(2)#2–Na(1)–W(1)#2	35.7(1)
O(7)–Na(1)–O(3)	99.2(3)	N(1)–Na(1)–W(1)#2	97.4(2)
O(7)–Na(1)–O(2)#2	90.9(2)	N(4)–Na(1)–W(1)#2	122.1(2)
O(3)–Na(1)–O(2)#2	104.7(2)	O(4)#2–Na(1)–W(1)#2	27.8(1)
O(7)–Na(1)–N(1)	163.4(3)	C(29)–N(1)–Na(1)	116.7(5)
O(3)–Na(1)–N(1)	95.6(2)	N(2)–C(28)–C(29)	113.1(6)
O(2)#2–Na(1)–N(1)	92.7(2)	N(1)–C(29)–C(28)	110.9(7)
O(7)–Na(1)–N(4)	80.0(3)	C(32)–O(7)–Na(1)	130.3(9)
O(3)–Na(1)–N(4)	96.7(2)	C(30)–N(4)–Na(1)	135.2(8)
O(2)#2–Na(1)–N(4)	157.8(2)	C(30)–C(31)–N(5)	120.7(1)
N(1)–Na(1)–N(4)	90.7(3)	C(31)–C(30)–N(4)	131.8(1)

Symmetry transformations used to generate equivalent atoms:
#1 $x, -y+3/2, z+1/2$; #2 $x, -y+3/2, z-1/2$.

parallel through π-π stacking interactions to form a chiral one-dimensional chain. The chiral chain couples with its enantiomer chain, linked by cations to constitute a meso-bilayer-chain. Two neighboring meso-bilayer units align anti-parallel at 0.56 nm to constitute an anti-parallel-aligned-double-meso-bilayer unit of 3.01 nm, and the double-meso-bilayer as repeating unit in crystal lattice assemble the multi-lamellar structure figure 2(b). The structure was stabilized by π-π stacking interactions and hydrogen bonds (table 5). The 1,3-propylenediamines are bridges that link the chains through $\mathrm{O}\cdots\mathrm{H{-}N}$ (2.17 Å, 145°), $\mathrm{N(4)}\cdots\mathrm{H(2B){-}N(2)}$ (2.07 Å, 178°),

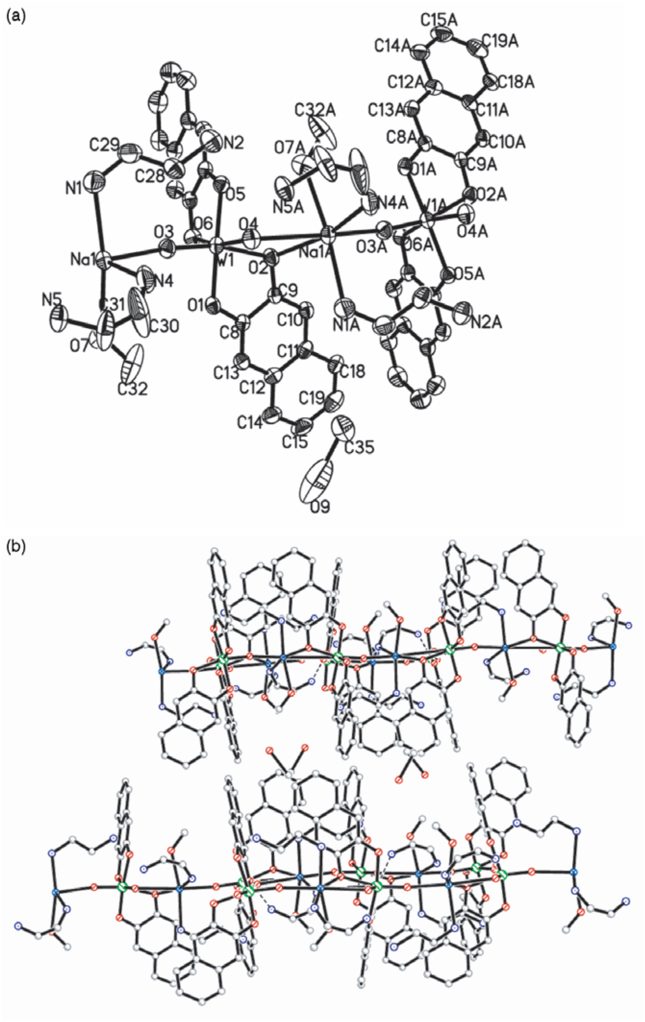

Figure 1. (a) Molecular structure of **1**; thermal ellipsoids have been drawn at the 30% probability level. (b) Packing diagram of **1** along the c-axis.

N(4)···H(3B)–N(3) (2.00 Å, 154°) and O(3)···H(3C)–N(3) (2.26 Å, 146°) hydrogen bonds (figure 2c).

3.4. *Discussion and analysis*

From the molecular structures, we can see clearly that complex **1** is a one-dimensional chain-like structure with Na in the structure, while **2** is a discrete monomer without Na. Two *cis*-dioxotungsten complexes have been synthesized in the same reaction conditions, except that protonated ethylenediamine was used in reaction **1** and 1,3-propylenediamine in reaction **2**. Therefore, the difference in structures arose from hindrance of 1,3-propylenediamine by three (–CH_2–) units.

Table 4. Selected bond lengths (Å) and angles (°) for **2**.

W(1)–O(4)	1.738(4)	C(7)–O(5)	1.342(6)
W(1)–O(3)	1.749(5)	C(8)–O(6)	1.311(6)
W(1)–O(5)	1.979(4)	C(6)–O(4)	1.422(1)
W(1)–O(2)	1.988(4)	C(22)–N(1)	1.453(1)
W(1)–O(6)	2.132(4)	C(23)–N(2)	1.430(1)
W(1)–O(1)	2.138(4)	C(24)–N(4)	1.455(8)
C(1)–O(2)	1.342(6)	C(26)–N(3)	1.468(7)
C(2)–O(1)	1.330(6)		
O(4)–W(1)–O(3)	101.8(2)	O(5)–W(1)–O(1)	89.0(2)
O(4)–W(1)–O(5)	90.8(2)	O(2)–W(1)–O(1)	75.6(2)
O(3)–W(1)–O(5)	102.7(2)	O(6)–W(1)–O(1)	81.3(2)
O(4)–W(1)–O(2)	104.2(2)	C(1)–O(2)–W(1)	119.5(3)
O(3)–W(1)–O(2)	90.1(2)	C(2)–O(1)–W(1)	115.5(3)
O(5)–W(1)–O(2)	158.1(2)	C(7)–O(5)–W(1)	119.3(3)
O(4)–W(1)–O(6)	163.8(2)	C(8)–O(6)–W(1)	114.7(3)
O(3)–W(1)–O(6)	90.7(2)	N(1)–C(22)–C(21)	115.9(8)
O(5)–W(1)–O(6)	76.2(2)	N(2)–C(23)–C(21)	118.3(7)
O(2)–W(1)–O(6)	86.1(2)	N(4)–C(24)–C(25)	113.5(5)
O(4)–W(1)–O(1)	89.0(2)	N(3)–C(26)–C(25)	115.0(5)
O(3)–W(1)–O(1)	163.9(2)		

In **1**, the Na connected to W atom through an O bridge and ethylenediamine coordinates with Na. In **2**, the hindrance of 1,3-propylenediamine molecular was bigger, preventing Na atom connecting with the W atom through an O bridge. Therefore, **2** is a monomer and 1,3-propylenediamine alone compensates the charge; only hydrogen bonds exist in the packing of **2**.

4. UV spectrum analysis

The UV spectrum of **1** and **2** in distilled water are shown in figure 3.

The UV spectrum of **1** exhibits five absorption bands at 228, 270, 309, 322 and 336 nm, while **2** shows bands at 241, 270, 310, 324 and 336 nm. Naphthalene has three absorption bands, E_1, E_2 and B. The absorption at 228 nm for **1** is the E_1 band, 270 nm E_2 and 309 nm B (241 nm, 270 nm and 310 nm for **2**). Strong absorptions at 324 nm and 322 nm, respectively, were assigned to the n-π^* transition of terminal O atom and naphthalene.

Tungsten(VI) has no d-electrons; therefore, absorptions at 336 nm for **1** and 336 nm for **2** are assigned to a ligand to metal charge transfer (LMCT) from the 2,3-dihydroxynaphthalene oxygen to an empty d-orbital of tungsten(VI).

Supporting information available

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center (CCDC), CCDC No. 608803 for **1** and

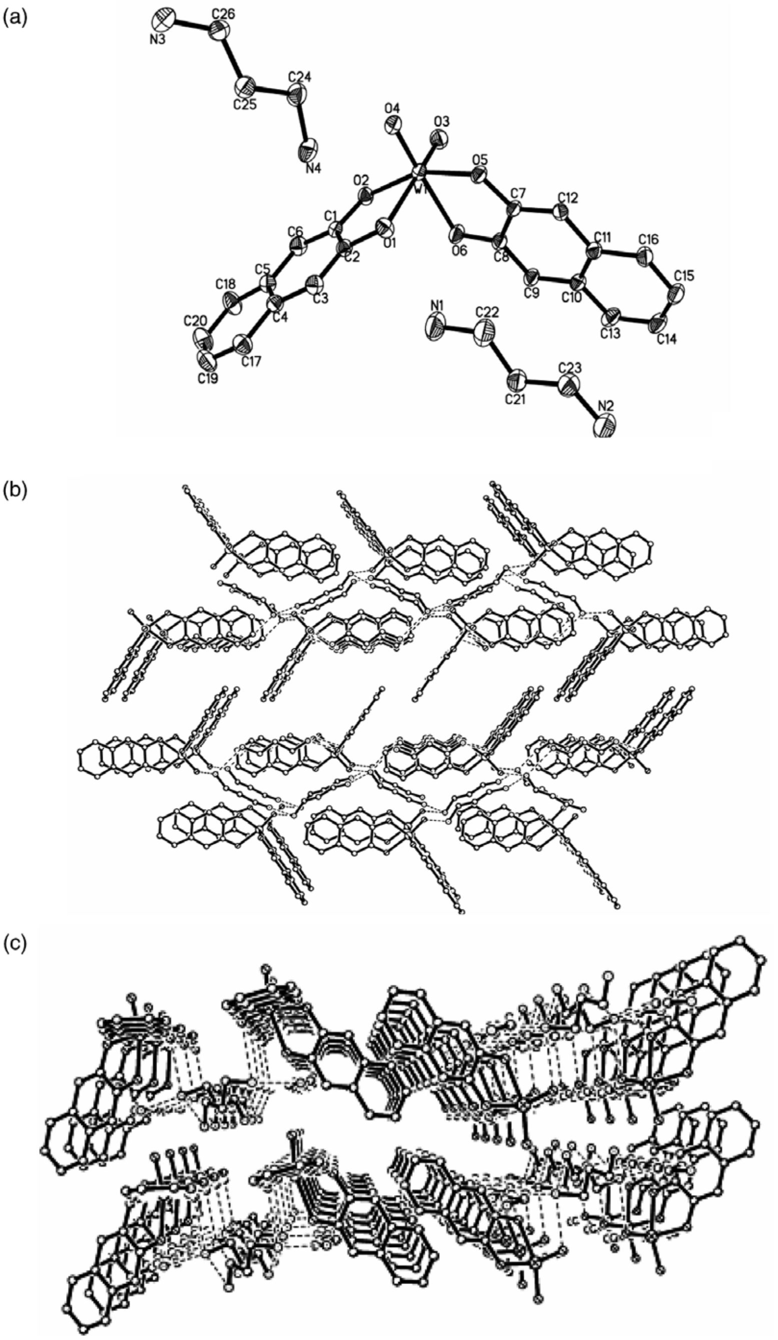

Figure 2. (a) Molecular structure of **2**; thermal ellipsoids have been drawn at the 30% probability level. (b) Packing diagram of **2** along the *a*-axis. (c) Packing diagram of **2** along the *c*-axis.

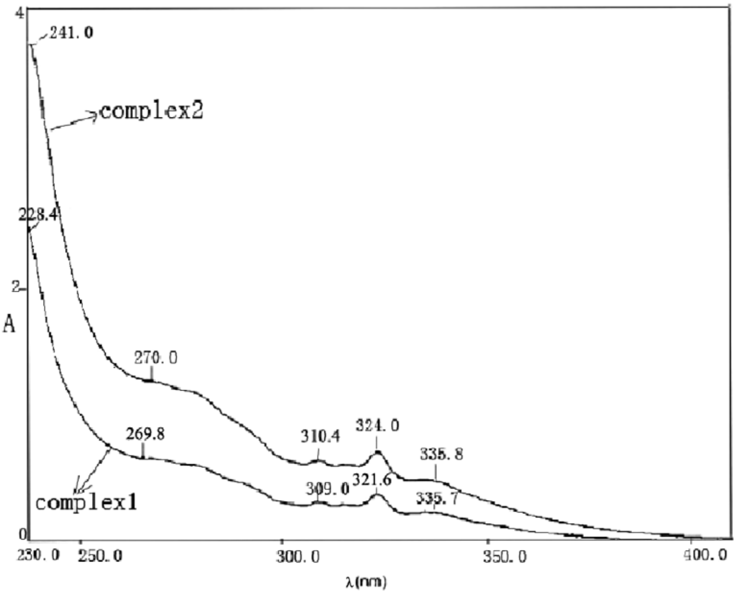

Figure 3. Absorption spectra of **1** and **2** in disstilled water.

Table 5. Hydrogen-bonding geometry (Å, °) of **2**.

Donor–H ··· Acceptor	D–H	H ··· A	D ··· A	D–H ··· A
N(1)–H(1B) ··· O(1)	0.89	2.17	2.941(9)	145
N(1)–H(1C) ··· O(3)	0.89	2.08	2.900(1)	154
N(2)–H(2B) ··· N(4)	0.89	2.07	2.961(8)	178
N(3)–H(3B) ··· N(4)	0.89	2.00	2.828(8)	154
N(3)–H(3C) ··· O(3)	0.89	2.26	3.038(8)	146
N(3)–H(3C) ··· O(4)	0.89	2.59	3.368(7)	147
N(4)–H(4A) ··· N(3)	0.89	1.99	2.828(8)	156
N(4)–H(4B) ··· O(1)	0.89	2.55	3.185(7)	129
N(4)–H(4B) ··· O(4)	0.89	2.04	2.892(7)	159
N(4)–H(4C) ··· N(2)	0.89	2.08	2.961(8)	168
C(3)–H(3) ··· O(3)	0.93	2.53	3.429(8)	163
C(26)–H(26A) ··· O(1)	0.97	2.60	3.435(8)	144

CCDC No. 624291 for **2**. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ (Fax: 044-1223-336-033 or E-mail: deposit@ccdc.cam.ac.uk).

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