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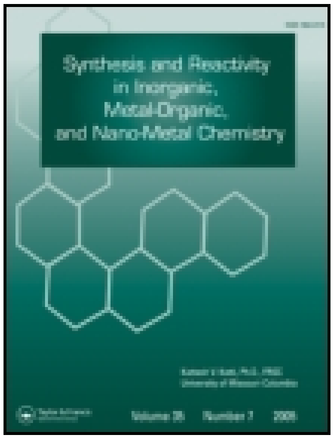

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Synthesis, Crystal Structure, and Properties of Two Novel Complexes Based on Zaltoprofen Ligand

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Synthesis, Crystal Structure, and Properties of Two Novel Complexes Based on Zaltoprofen Ligand

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Two novel metal-organic coordination complexes [Cd(Zaltoprofen)$_2$(H_2O)$_3$](H_2O) (1), [Zn(Zaltoprofen)(H_2O)]$_n$ (2) (where zaltoprofen = 5-(1-carboxyethyl)-2-(phenylthio)phenylacetic acid) have been synthesized under normal conditions and hydrothermal conditions, respectively. And characterized by single crystal X-ray diffraction, elemental analysis, spectral method (IR), powder X-ray diffraction (XRD), and fluorescent properties. Compound 1 crystallizes in the monoclinic *Cc* space group while compound 2 in the monoclinic system with space group *P2$_1$/c*. [Cd(Zaltoprofen)$_2$(H_2O)$_3$](H_2O) (1) features 0-D structural, the hydrogen-bonds play a vital role in determining the crystal packing and construction of the extended 3-D supramolecular network. [Zn(Zaltoprofen)(H_2O)]$_n$ (2) displays a 2D net with alternately arranged left- and right-handed helical chains. The O–H···O hydrogen bonding play an important role in stabilizing the structure.

Supplemental materials are available for this article. Go to the publisher's online edition of* Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry *to view the supplemental file.

Keywords Cd(II) complex, single-crystal structure, zaltoprofen, Zn(II) complex

INTRODUCTION

In recent years, the low-dimensional coordination polymers have received much attention owing to their intriguing structural features and potential as functional materials different from those of 3-D coordination polymers.[1–4] A particularly interesting and challenging area is the synthesis of helical coordination polymers and exploration of their potential utility in asymmetric catalysis and nonlinear optics.[5–12] Metal–organic helical structures containing single-, double-, and multistranded helices[13–21] have been constructed; the chemistry of coordination polymers with helical structures has been reviewed.[22,23] Hydrogen-bonding plays an important role in the fabrication of the supramolecular architectures.

In this respect, the influence of ligand spacers of flexible bridging ligands on framework formation of their coordination polymers has widely been documented.[24–28] Zaltoprofen is one of non-steroidal anti-inflammatory drugs that exhibit favorable anti-inflammatory, analgesic, and antipyretic properties. Most anti-inflammatory drugs are carboxylic acids in which carboxylate is available for metal–ligand interaction. Much work has focused on interactions of transition metal ions with non-steroidal anti-inflammatory drugs such as ibuprofen, fenoprofen.[29,30] But to our knowledge, using Zaltoprofen to construct metal–organic frameworks has not been reported. As a multi-functional ligand, Zaltoprofen has attracted our attention on the basis of the following considerations. The multicarboxylates are capable of functioning as hydrogen bond donors and/or acceptors. The carboxyl is always deprotonated to compensate the charge of the metal ions, and it may allow for diversity in the coordination mode. The flexible Zaltoprofen building blocks considered as V-shaped dicarboxylate may generate helical chains.

As a continuation of our research, we report synthesis, crystal structure, elemental analyses, IR spectrum, powder XRD, and fluorescent properties of two novel complexes [Cd(Zaltoprofen)$_2$(H_2O)$_3$](H_2O) (**1**), [Zn(Zaltoprofen)(H_2O)]$_n$ (**2**), which are constructed and stabilized the framework via Hydrogen-bonding. Photoluminescence properties of complexes in DMF/EtOH have also been investigated below in detail.

EXPERIMENTAL

All chemicals were commercial materials of analytical grade and used without purification. Elemental analysis for C, H, and N was carried out on a Perkin-Elmer 2400 II elemental analyzer.

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TABLE 1
Experimental data for complex **1** and **2**

	1	2
Empirical formula	$C_{34}H_{38}CdO_{12}S_2$	$C_{17}H_{16}ZnO_5S$
Formula weight	815.16	397.73
Temperature	296(2) K	296(2) K
Wavelength	0.71073 Å	0.71073 Å
Crystal system, space group	Monoclinic, *Cc*	Monoclinic, $P2_1/c$
a (Å)	38.520(2)	18.492(2)
b (Å)	11.423(3)	5.436(1)
c (Å)	8.145(3)	19.104(3)
β (°)	90.703(1)	120.688(1)
Volume (Å^3)	3583.8(2)	1651.3(5)
Z	4	4
$D_{calcd.}$ (Mg·m^{-3})	1.511	1.600
Absorption coefficient (mm^{-1})	0.787	1.637
F(000)	1672	816
Crystal size	0.23 × 0.22 × 0.20	0.38 × 0.35 × 0.33
θ range for data collection (°)	1.06–25.00	2.13–25.00
Reflections collected	8971	8436
Unique reflections	4598 [$R_{(int)} = 0.0421$]	2910 [$R(int) = 0.0284$]
Completeness to $\theta = 25.00$	99.6%	99.9%
Absorption correction	Semiempirical	Semiempirical
Max. and min. transmission	0.854 and 0.834	0.583 and 0.542
Data / restraints / parameters	4598 / 39 / 390	2910 / 7 / 218
Goodness-of-fit on F^2	1.093	1.029
R indices [$I > 2\sigma(I)$]	$R_1 = 0.0905$, $wR_2 = 0.2465$	R1 = 0.0343, wR2 = 0.0868
R indices (all data)	$R_1 = 0.0974$, $wR_2 = 0.2533$	R1 = 0.0430, wR2 = 0.0945
Largest diff. peak and hole (e.Å^{-3})	3.118 and 1.636	0.748 and −0.475

The FT-IR spectrum was obtained on a PE Spectrum One FT-IR Spectrometer Fourier transform infrared spectroscopy in the 4000-400 cm^{-1} regions, using KBr pellets. Powder X-ray diffraction patterns were obtained using a pinhole camera (Anton Paar) operating with a point focused Ni-filtered Cu Kα radiation in the 2θ range from 5° to 50° with a scan rate of 0.08° per second.

Synthesis of [Cd(Zaltoprofen)$_2$(H$_2$O)$_3$](H$_2$O) (1)

$CdSO_4 \cdot 8/3H_2O$ (0.2565 g, 0.1 mmol), Zaltoprofen (0.1582 g, 0.5 mmol) were dissolved in the mixture of 10 mL CH_3CH_2OH and 10 mL H_2O. Then an aqueous solution of sodium hydroxide was added dropwise with stirring to adjust the pH value of the solution being 6. The mixture was kept stirring for 6 h at 74°C and then filtered. The filtrate was kept at room temperature and a few days later X-ray quality colorless block-shaped single crystals were obtained. The colorless block crystals suitable for X-ray diffraction were isolated directly, Yield: 74%, based on Cd. Anal. Calcd. for $C_{34}H_{38}CdO_{12}S_2$ (%): C,50.09; H, 4.70. Found: C, 50.27; H, 4.86. IR data (KBr pellets, cm^{-1}): 3417 (s), 2981 (w), 1665 (vs), 1542 (vs), 1476 (m), 1396 (m), 1302 (m), 1187 (w), 1072 (w), 1023 (w), 944 (w), 906 (w), 831 (w), 808 (w), 739 (m), 689 (m), 623 (w), 540 (w), 490 (w), 471 (w).

Synthesis of [Zn(Zaltoprofen)(H$_2$O)]$_n$ (2)

The reagents of $ZnCl_2 \cdot 6H_2O$ (0.2975 g, 1 mmol), Zaltoprofen (0.1582 g, 0.5 mmol) 20 mL mixed solvent of CH_3CH_2OH /H_2O (volume ratio 1:3) were sealed in a 30 mL Teflon-lined stainless reactor, an aqueous solution of sodium hydroxide was added dropwise with stirring to adjust the pH value of the solution being 6 and the mixture was stirred for 0.5 h, then kept under autogenous pressure at 160°C for 24 h. The resulting reaction mixture was slowly cooled to room temperature at a rate of 5°C per hour. The colorless block crystals suitable for X- ray diffraction were isolated directly, Yield: 58%, based on Zn. Anal. Calcd. for $C_{17}H_{16}ZnO_5S$ (%): C, 51.33; H, 4.05. Found: C, 51.48; H, 4.27. IR data (KBr pellets, cm-1): 3432 (s), 2977 (w), 1615 (vs), 1565 (vs), 1425 (w), 1383 (s), 1284 (w), 1254 (w), 1065 (w), 1023 (w), 904 (w), 837 (w), 802 (m), 736 (m), 689 (m), 651 (w), 469 (w).

TABLE 2
The selected bond lengths (Å) and angles (°)

Complex 1			
Cd(1)-O(5)	2.262(1)	Cd(1)-O(7)	2.225(2)
Cd(1)-O(11)	2.18(2)	Cd(1)-O(1)	2.346(1)
Cd(1)-O(10)	2.352(1)	Cd(1)-O(2)	2.442(1)
Cd(1)-O(6)	2.520(1)	O(11)-Cd(1)-O(7)	98.5(7)
O(11)-Cd(1)-O(5)	83.8(7)	O(7)-Cd(1)-O(5)	132.8(5)
O(11)-Cd(1)-O(1)	86.1(6)	O(7)-Cd(1)-O(1)	89.8(5)
O(5)-Cd(1)-O(1)	137.3(5)	O(11)-Cd(1)-O(10)	168.4(7)
O(7)-Cd(1)-O(10)	92.1(5)	O(5)-Cd(1)-O(10)	92.4(5)
O(1)-Cd(1)-O(10)	89.3(5)	O(11)-Cd(1)-O(2)	90.5(7)
O(7)-Cd(1)-O(2)	142.4(5)	O(5)-Cd(1)-O(2)	84.3(5)
O(1)-Cd(1)-O(2)	54.3(4)	O(10)-Cd(1)-O(2)	78.2(4)
O(11)-Cd(1)-O(6)	111.8(7)	O(7)-Cd(1)-O(6)	53.3(5)
O(5)-Cd(1)-O(6)	81.9(5)	O(1)-Cd(1)-O(6)	139.8(5)
O(10)-Cd(1)-O(6)	78.3(5)	O(2)-Cd(1)-O(6)	152.1(3)
Complex 2			
Zn(1)-O(1)	1.947(2)	Zn(1)-O(4)#1	1.957(2)
Zn(1)-O(5)	1.971(2)	Zn(1)-O(3)#2	1.982(2)
O(1)-Zn(1)-O(4)#1	95.87(9)	O(1)-Zn(1)-O(5)	125.97(9)
O(4)#1-Zn(1)-O(5)	111.89(9)	O(1)-Zn(1)-O(3)#2	102.82(9)
O(4)#1-Zn(1)-O(3)#2	115.24(9)	O(5)-Zn(1)-O(3)#2	105.25(9)

Symmetry transformations used to generate equivalent atoms: for complex **1**: #1 −x, −y, −z + 1; for complex **2**: #1 −x, −y + 1, −z #2 x, −y + 3/2, z − 1/2.

Crystal Structure Determination

Single crystal of the complex was mounted on glass fibers and measured on a Bruker SMART CCD area detector at 298 K using graphite monochromated Mo Ka radiation (λ = 0.71073 Å). Empirical absorption corrections were applied using the SADABS program.[31] The complexes were solved by Patterson method and direct method, respectively. And refined by full-matrix least squares on F^2 using the SHELXTL program.[32] All non-hydrogen atoms were refined anisotronically. A summary of the crystallographic data and structure refinement is shown in Table 1, selected bond lengths and angles of the complexes are listed in Table 2.

RESULTS AND DISCUSSION

Description of the Structure

Complex **1** crystallizes in the monoclinic space group of *Cc*. In complex **1** the asymmetric unit contains one Cd(II) ions, four water molecules, and two half deprotonated Zaltoprofen$^-$ ligands. The Cd(II) center is seven-coordinated by four oxygen atoms (O1, O2, O6, O7) of the carboxylate from two Zaltoprofen$^-$ ligands and one oxygen atoms (O5) from one water molecule composing the equatorial plane, and two oxygens atoms (O10, O11) from two water molecules at the axial sites (Figure 1). The Cd-O bond distances vary from 2.18(2) to 2.520(1) Å. One carboxylate group adopts a μ^1-η^1:η^1 chelating coordination mode to connect one Cd(II) atom while the other carboxylate group is not coordinated. There are hydrogen bonds among all the oxygen atom (O3, O4, O8, O9) of the nondeprotonated carboxyl group, the oxygen atoms (O1, O2, O6, O7) of the coordinated carboxyl group, the oxygen atoms (O5, O10, O11) of the coordinated water molecule and the oxygen atom (O12) of the uncoordinated water molecule, determining the crystal

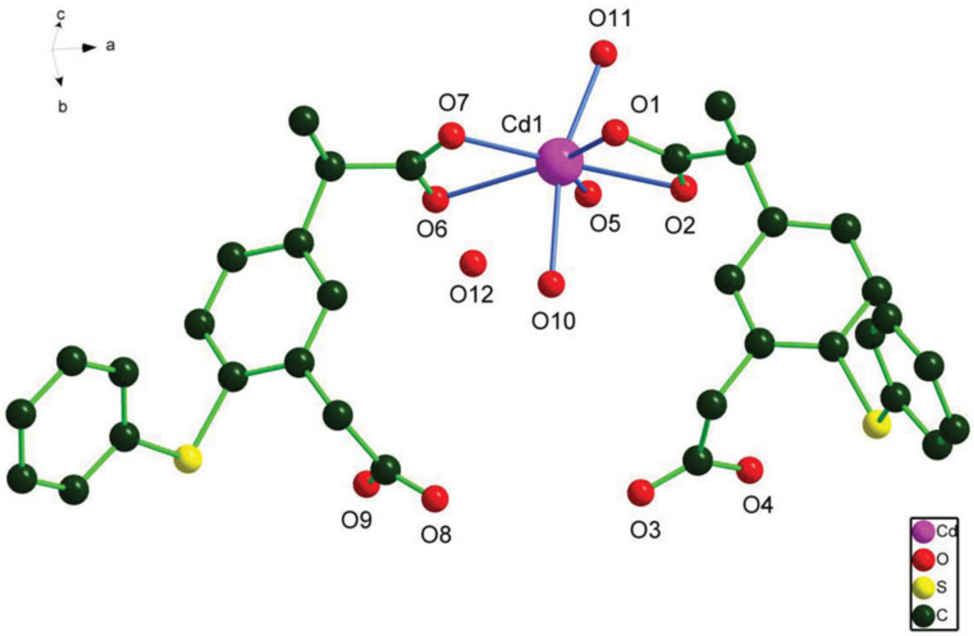

FIG. 1. The coordination environment of Cd(II) ion of complex **1**, All the hydrogen atoms are omitted for clarity. (color figure available online).

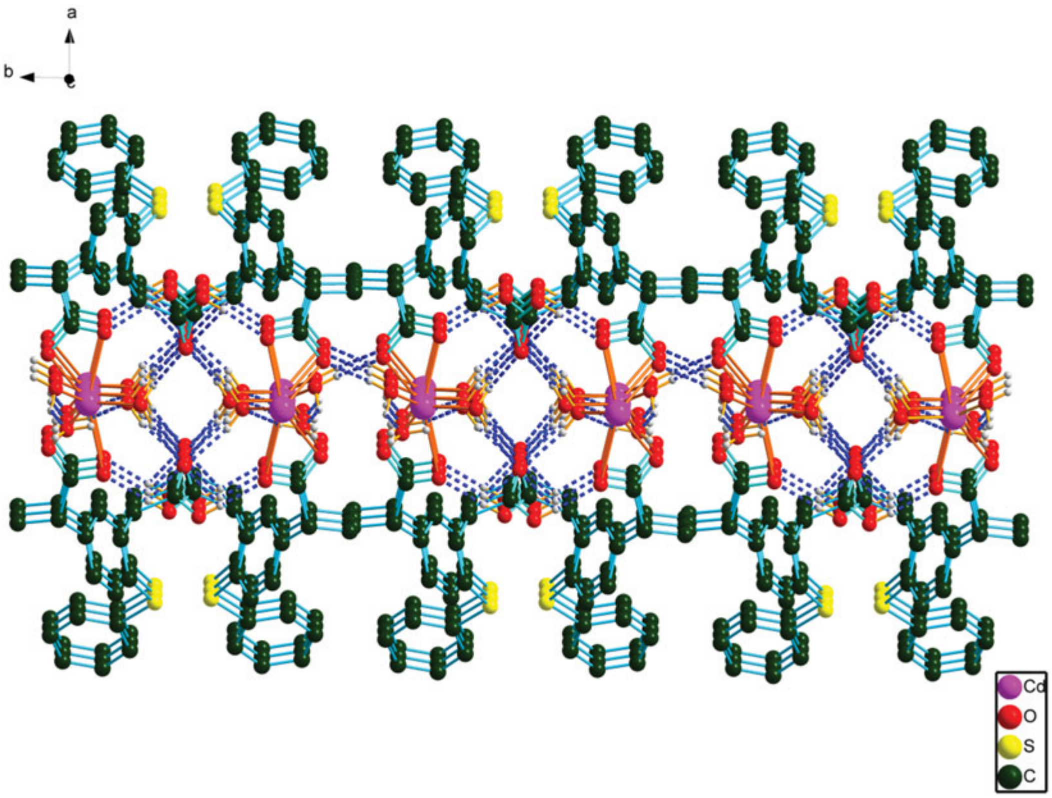

FIG. 2. The 3D framework of complex **1** via H-bonding. The unnecessary hydrogen atoms are omitted for clarity. (color figure available online).

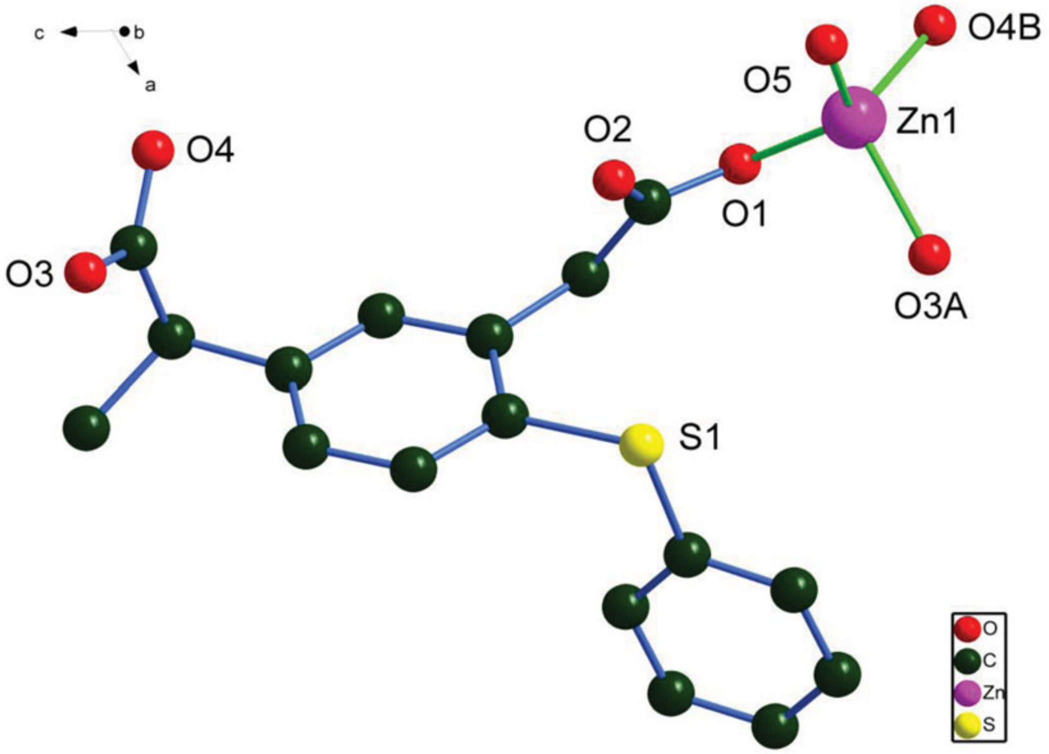

FIG. 3. The coordination environment of Zn(II) ion of complex **2**, All the hydrogen atoms are omitted for clarity. (color figure available online).

TABLE 3
Hydrogen-bonding geometries (Å) for **1** and **2**

D–H. . .A	Symmetry codes	D-H	H. . .A	D. . .A	D–H. . .A
Complex **1**					
O9-H9A. . .O6	x, −y + 2, z − 1/2	0.82	1.772	2.588	173.01
O4-H4A. . .O2	x, −y + 2, z − 1/2	0.82	1.805	2.619	171.49
O12-H12B. . .O10		0.85	2.27	2.97(2)	139.9
O12-H12A. . .O7		0.85	2.56	3.36(2)	157.9
O11-H11B_a. . .O1	x, −y + 1, z + 1/2	0.85	2.12	2.77(2)	132.9
O11-H11A_a. . .O12	x, y, z + 1	0.85	2.03	2.72(2)	138.4
O10-H10D. . .O8	x, −y + 2, z + 1/2	0.85	1.97	2.80(1)	168.2
O10-H10C. . .O3	x, −y + 2, z + 1/2	0.85	1.99	2.82(1)	167.0
O5-H5B. . .O3	x, y, z + 1	0.85	2.06	2.64(1)	124.5
O5-H5A. . .O8	x, y, z + 1	0.85	2.18	2.72(1)	121.5
Complex **2**					
O5-H5C. . .O2	−x, −y + 2, −z	0.85	1.82	2.663(3)	169.5
O5-H5B. . .O1	x, y + 1, z	0.85	1.95	2.786(3)	169.9

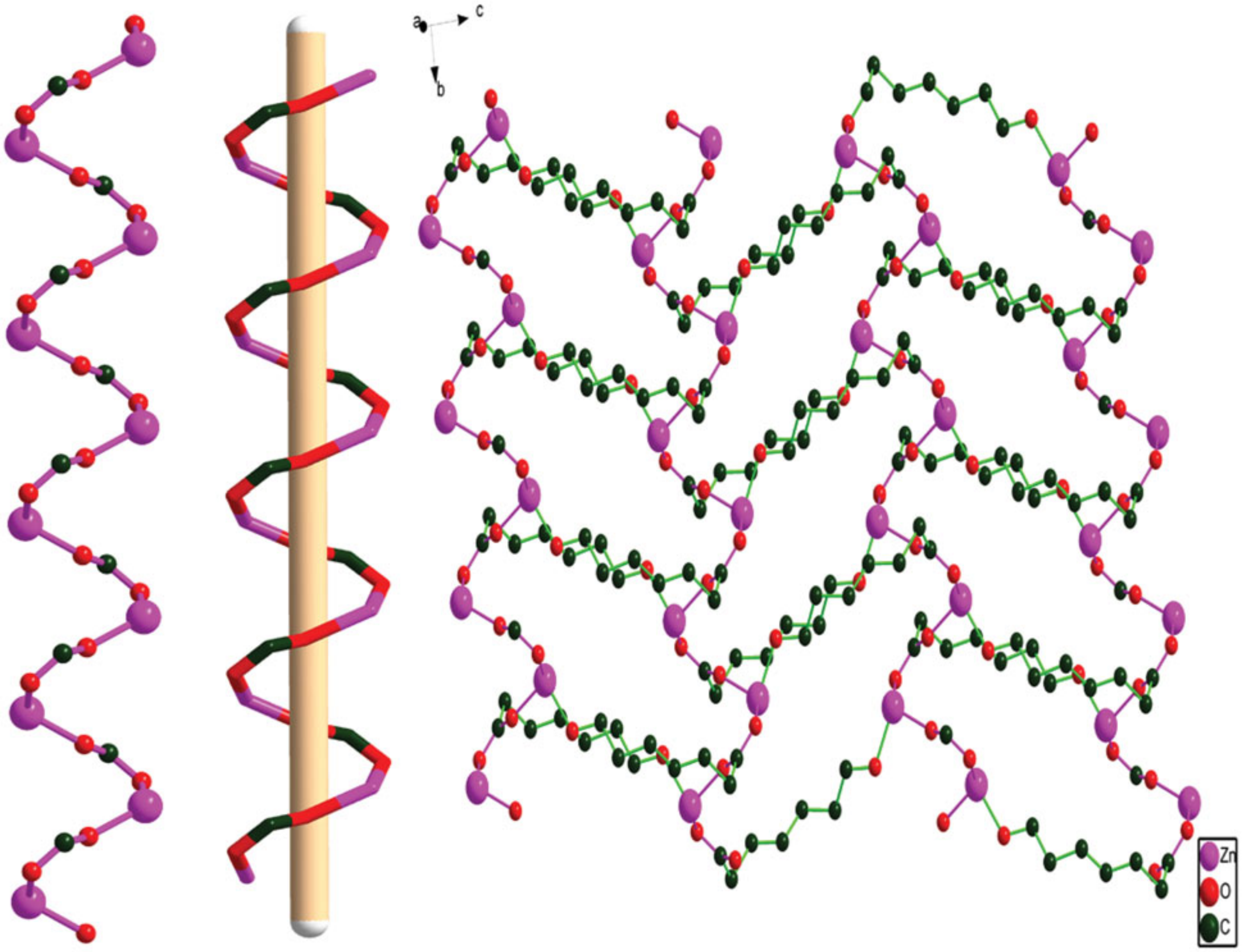

FIG. 4. View of the 2D Zn(II)/Zaltoprofen^{2-} net constructed by alternately left- and right-handed helical chains of complex **2**. Unnecessary atoms are omitted for clarity. (color figure available online).

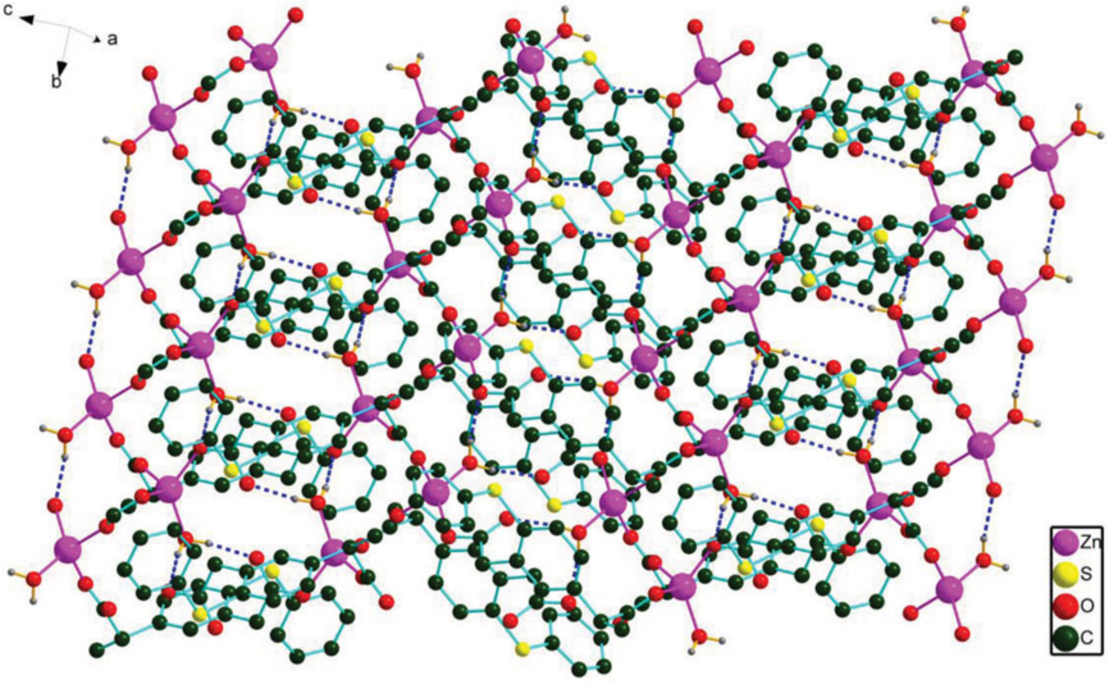

FIG. 5. View of the O–H···O hydrogen bonds in **2**. All unnecessary hydrogens are omitted for clarity. (color figure available online).

packing and construction of the extended 3-D supramolecular network (Figure 2 and Table 3).

The single-crystal X-ray diffraction analysis reveals that polymer **2** crystallizes in the monoclinic system, space group *P2₁/c*. The asymmetric unit consists of one Zn(II) ion, one water molecule and one completely deprotonated Zaltoprofen^{2-} ligand. As showed in Figure 3, the coordination environment around the Zn(II) center is best portrayed as a distorted [ZnO4] tetrahedron geometry, ligated by three oxygen donors (O1, O3A, O4B) belonging to three Zaltoprofen^{2-} ligands and one oxygen donor (O5) from one water molecule. The bond lengths of Zn-O are between 1.947(2) and 1.982(2) Å. In **2**, two carboxylate groups of the V-shape ligand Zaltoprofen^{2-} adopt different coordination modes: one carboxylate group adopts μ^1-η^1:η^0 mode and the other adopts μ^2-η^1: η^1-bridging mode. The Zaltoprofen^{2-} ligand acts as a μ3-bridge linking Zn(II) ions to produce a 2D net with alternately arranged left- and right-handed helical chains (Figure 4). The O–H···O hydrogen bonding play an important role in stabilizing 2D layer (Figure 5 and Table 3).

IR Spectra

Dried samples of complex was characterized by IR spectroscopy. In the IR spectra of two complexes, the strong and broad absorption bands at about 3500-3230 cm^{-1} are attributed to the symmetric O–H stretching modes. The IR spectrum of free ligand shows strong bands of the carboxylate groups at 1697 cm^{-1}, which can be assigned as the ν(C=O) antisymmetric stretching vibrations. In complex **1**, the corresponding bands of the nondeprotonated carboxylate groups at 1665 cm^{-1} while the coordinated carboxyl group at 1542 cm^{-1}. The corresponding bands of the monodentate coordinated carboxylate groups at 1615 cm^{-1} as well as the bidentate coordinated carboxyl group at 1565 cm^{-1} for complex **2**. The shift to the lower position suggests that the relevant oxygen of the ligand coordinates to metal.

XRD Patterns

Simulated and experimental powder X-ray diffraction (PXRD) patterns of complexes **1** and **2** are shown in Figure S1. All the peaks in the recorded curves approximately match those in the simulated curves generated from single-crystal diffraction data, which confirms the phase purity of the as-prepared products. The difference in reflection intensity between the simulated and experimental patterns is due to a certain degree of preferred orientation of the powder samples during data collection.

Fluorescence Properties

Inorganic-organic hybrid coordination polymers, especially those with d^{10} metal centers, have been investigated for photoluminescent properties. Previous studies have shown that coordination polymers containing cadmium(II) and zinc(II) exhibit photoluminescent properties.[33–38] The fluorescence properties of the complexes were studied in DMF/EtOH (1/1) at room temperature (298 K) (Figure 6). The complexes **1** and **2**, upon excitation at the wavelengths where their two MLCT absorption maxima are observed, exhibit broad luminescent maxima at 738 nm (λex = 371 nm) for **1** and at 683 nm (λex = 344 nm) for **2** in DMF/EtOH at room temperature, which remain unchanged with

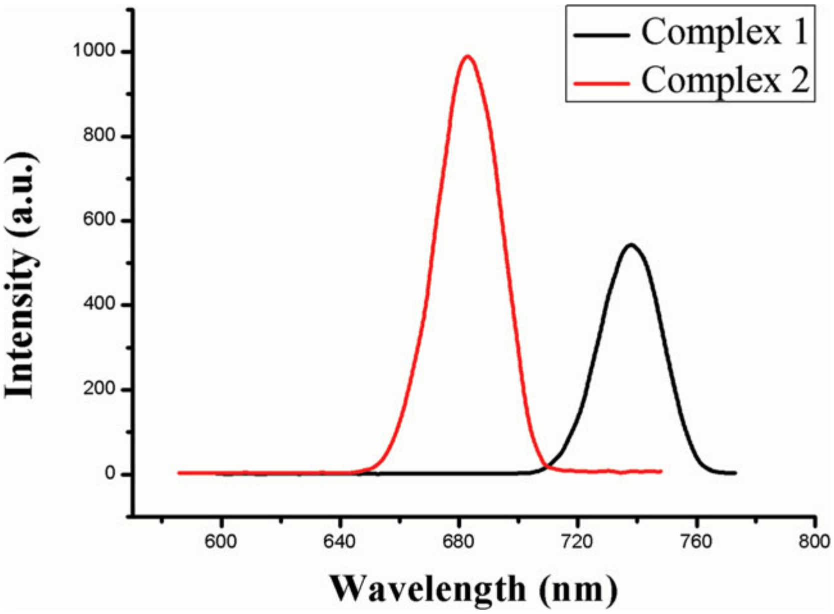

FIG. 6. Emission spectra of complex **1** (black) and **2** (red) in DMF/EtOH (1/1) recorded at room temperature. (color figure available online).

the energy of excitation wavelengths. For excitation wavelength between 320 and 480 nm, There is no obvious emission observed for free Zaltoprofen. Therefore, the fluorescent emissions in the coordination polymers may be proposed to originate from the coordination of Zaltoprofen anion to the metal atoms. The spectroscopic behavior of the complexes containing Zaltoprofen as a ligand is discussed in various spectroscopic transitions including ligand-centered and metal-ligand charge transfer.

CONCLUSION

On this work, we report the synthesis and characterization of two complexes [Cd(Zaltoprofen)$_2$(H$_2$O)$_3$](H$_2$O) and [Zn(Zaltoprofen)(H$_2$O)]$_n$ by X-ray diffraction analysis, elemental analysis, IR spectrum, powder X-ray diffraction, and fluorescence properties. The complex **1** is an 0–D structure while the complex **2** is 2–D net with alternately arranged left- and right-handed helical chains. The successful preparation of the complex manifest that the conformations and functions of hydrogen-bonding are important factors in influencing the nal architecture of Zaltoprofen coordination polymers. The presence of chelate ligands in transition metal dicarboxylate systems usually results in the formation of low–dimensional coordination polymers with the metal ions acting as nodes and the dicarboxylate ligands as connectors, especially 1–D chains, and the employment of a V–shaped dicarboxylate may generate zigzag and helical chains. The two polymers exhibit intense emissions, which appear to be potential hybrid inorganic–organic photoactive materials. Subsequent studies will be focused on the structures and properties of a series of coordination polymers constructed by Zaltoprofen with more organic ligands and other metal ions.

SUPPLEMENTARY MATERIALS

Further information about the crystallographic analysis is deposited in the "Cambridge Crystallographic Data Center" with registration number CCDC 907643 for **1** and 907642 for **2**. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, by e-mailing data deposit@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax +44(0)1223-336-033.

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