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Syntheses, structures and luminescent properties of a dimer and an one-dimensional chain coordination polymer with the flexible bis(triazole) and hydroxybenzoate ligands

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Abstract

Two novel complexes $[Zn(phba)_2(btp)]_2$ (1) and $[Cd(phba)_2(btp)(H_2O)]_n$ (2) have been synthesized by using 1,3-bis(1,2,4-triazol-1-yl)propane (btp) and 4-hydroxybenzoate (phba). 1 contains the neutral dimer $Zn_2(btp)_2$ metallacycle, while 2 forms the one-dimensional zigzag chain structure. 1 and 2 exhibit blue luminescent emissions at 402 and 394 nm, respectively, excited at 340 nm in the solid state at room temperature.

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1. Introduction

The design and construction of the coordination polymers and polynuclear complexes has attracted great attention in recent years for their potential application as well as the variety of architectures and topologies [1-9]. The selection of the ligands is undoubtedly the key point in the synthesizing of the coordination polymers and polynuclear complexes.

1,2,4-Triazole and its derivatives are very interesting ligands because they combine the coordination geometry of both pyrazole and imidazole with regard to the arrangement of their three heteroatoms. A large number of mononuclear, oligonuclear and polynuclear transition metal complexes of 1,2,4-triazole derivatives have been synthesized and characterized due to their magnetic properties and novel topologies [10–15]. However the complexes of the flexible bis(triazole) ligands have not been well exploited up to now [16–19]. Our interest is to study the coordination chemistry of 1,2,4-triazole and its derivatives together

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with their potential application in the material science. Recently, we reported the crystal structures of a series of transition metal coordination polymers with the flexible bis(triazole) ligands such as 1,2-bis(1,2,4-triazol-1-yl)eth-ane [20–23] and 1,4-bis(1,2,4-triazol-1-ylmethyl)benzene [24,25].

On the other hand, although salicylate has been widely employed for the synthesis of the coordination compounds [26,27]. 4-Hydroxybenzoate, the isomer of salicylate, was rarely studied to date [28]. The combination of the flexible ligand 1,3-bis(1,2,4-triazol-1-yl)propane (btp) and 4hydroxybenzoate (phba) can give rise to novel complexes. In the present paper, we report the preparation, crystal structure and luminescent properties of a dimer $[Zn(phba)_2(btp)]_2$ (1) and an one-dimensional zigzag chain coordination polymer $[Cd(phba)_2(btp)(H_2O)]_n$ (2).

2. Experimental

2.1. Materials and general methods

The flexible ligand 1,3-bis(1,2,4-triazol-1-yl)propane (btp) was synthesized according to the literature method

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[18]. All other reagents and solvents employed were commercially available and used as received without further purification. The C, H, N microanalyses were carried out with a Perkin-Elmer 240C analyser. IR spectra were obtained for KBr pellets on a Nicolet 170SX FT-IR spectrophotometer in the 4000–400 cm⁻¹ region. The luminescence measurements were carried out in the solid state at room temperature and the spectra were collected with a Perkin-Elmer LS50B spectrofluorimeter.

2.2. Preparation of the complexes

2.2.1. $[Zn(phba)_2(btp)]_2(1)$

A 25 mL water/MeOH solution (1:1 v/v) of $Zn(NO_3)_26H_2O$ (0.5 mmol) was added to one leg of an "H-shaped" tube, and a 25 mL water/MeOH (1:1 v/v) solution of 1,3-bis(1,2,4-triazol-1-yl)propane (btp) (0.5 mmol) and sodium 4-hydrogenbenzoate (1.0 mmol) was added to the other leg of the tube. Colourless crystal 1 (yield: 37%) were obtained after about two months. Anal. Calcd. for C₄₂H₄₀N₁₂O₁₂Zn₂ (1): C, 48.71; H, 3.89; N, 16.23%. Found: C, 48.57; H, 3.74; N, 16.01. IR data (cm⁻¹): 3241 m, 3141 m, 1605 s, 1536 s, 1505 m, 1454 m, 1397 s, 1273 m, 1243 m, 1219 m, 1165w, 1135 m, 1095w, 996w, 857w, 787 m, 671w, 641 m, 417w.

2.2.2. $[Cd(phba)_2(btp)(H_2O)]_n$ (2)

The synthesis procedure of **2** was similar to the synthesis of **1**, except that Cd(NO₃)₂.6H₂O was instead of Zn(NO₃)₂6H₂O. Anal. Calcd. for C₂₁H₂₂CdN₆O₇ (**2**): C, 43.28; H, 3.80; N, 14.42%. Found: C, 43.21; H, 3.74; N, 14.35. IR data (cm⁻¹): 3386s, 3118vs, 1698w, 1598s, 1528s, 1505s, 1443m, 1389s, 1281s, 1243s, 1165m, 1104m, 1034w, 934w, 857m, 795m, 749m, 702w, 656m, 625m, 548w.

2.3. X-ray crystallography

All data collections were performed at the temperature of 193(2) K on a Rigaku Mercury CCD diffractometer with the ω -scan technique. The structures were solved by direct methods and refined with the full-matrix least-squares technique using the SHELXS-97 and SHELXL-97 programs [29]. Anisotropic thermal parameters were assigned to all non-hydrogen atoms. The hydrogen atoms from btp and phba ligands were generated geometrically. The hydrogen atoms from water molecules were obtained from difference Fourier maps. The crystallographic data for 1 and 2 are listed in Table 1. The selected bond lengths and angles for 1 and 2 are listed in Table 2.

3. Results and discussion

In the IR spectra, the conspicuous carboxylate stretching at 1605, 1505, 1454 and 1397 cm⁻¹ in **1**, 1598, 1505, 1443 and 1389 cm⁻¹ in **2**. **1** and **2** both present two groups of the antisymmetric $v_a(COO)$ and symmetric stretching

Table 1	
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Crystallographic data for 1 and 2

Empirical formula	C42H40N12O12Zn2	$C_{21}H_{22}CdN_6O_7$
Formula weight	1035.60	582.85
Temperature (K)	193(2)	193(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/c$	$P\overline{1}$
a (Å)	10.750(2)	9.9743(12)
b (Å)	19.234(3)	11.0238(7)
<i>c</i> (Å)	11.432(3)	11.2144(9)
α	90	66.090(8)
β (°)	114.071(4)	77.431(9)
γ	90	83.062(9)
$V(Å^3)$	2158.3(7)	1099.51(17)
Ζ	2	2
F(000)	1064	588
ρ [g.cm ⁻³]	1.594	1.760
μ [mm ⁻¹]	1.191	1.051
Crystal size [mm]	$0.38 \times 0.31 \times 0.19$	$0.40 \times 0.28 \times 0.15$
θ range for data collection (°)	3.05–25.34	3.17–25.35
Index ranges	$-12 \leq h \leq 12$,	$-10 \leq h \leq 12$,
-	$-22 \leqslant k \leqslant 23,$	$-12 \leqslant k \leqslant 13$,
	$-13 \leqslant 1 \leqslant 12$	$-13 \leq l \leq 13$
Refinement method	Full-matrix least-	Full-matrix least-
	squares on F^2	squares on F^2
Independent reflections	3940[R(int) = 0.0337]	3981[R(int) = 0.0180]
Parameter	308	324
Max. and min. transmission	0.8053 and 0.6603	0.8582 and 0.6785
Goodness of fit S	1.095	1.014
Final R_1 and wR_2 [$I > 2\sigma(I)$]	0.0366 and 0.0855	0.0204 and 0.0519
R_1 and wR_2 indices (all data)	0.0419 and 0.0884	0.0225 and 0.0532
Largest diff. Peak and hole (e $Å^{-3}$)	0.330 and -0.471	0.578 and -0.444

Table 2

Selected bond lengths (Å) and angles (°) for 1 and 2

1			
Zn(1)-O(1)	2.153(2)	Zn(1)-O(2)	2.152(2)
Zn(1)-O(4)	1.9839(17)	Zn(1)-N(3)	2.066(2)
Zn(1)-N(6A)	2.064(2)		
O(2)-Zn(1)-O(1)	60.74(8)	O(4)-Zn(1)-O(1)	100.54(8)
O(4)-Zn(1)-O(2)	161.02(8)	N(6A)-Zn(1)-O(1)	143.96(8)
N(6A)-Zn(1)-O(2)	91.61(8)	O(4)-Zn(1)-N(6A)	103.95(8)
N(3)-Zn(1)-O(1)	107.80(8)	N(3)-Zn(1)-O(2)	92.86(9)
O(4)-Zn(1)-N(3)	96.37(8)	N(6A)-Zn(1)-N(3)	95.36(8)
2			
Cd(1)–O(1)	2.4841(14)	Cd(1)–O(2)	2.3054(14)
Cd(1)-O(4)	2.2916(14)	Cd(1)–O(7)	2.4502(17)
Cd(1)-N(3)	2.2574(16)	Cd(1)-N(6A)	2.2572(16)
O(2)-Cd(1)-O(1)	54.57(5)	N(3)-Cd(1)-O(1)	142.67(5)
N(6A)-Cd(1)-O(1)	95.41(5)	N(3)-Cd(1)-O(2)	91.03(5)
N(6A)-Cd(1)-O(2)	149.95(5)	N(3)-Cd(1)-N(6A)	117.27(6)
O(4)-Cd(1)-O(7)	159.74(6)	O(4)-Cd(1)-O(1)	82.38(5)
O(4)-Cd(1)-O(2)	88.28(5)	N(3)-Cd(1)-O(4)	113.75(6)
N(6A)-Cd(1)-O(4)	89.07(6)	O(7)-Cd(1)-O(1)	79.28(6)
O(2)-Cd(1)-O(7)	88.08(6)	N(3)-Cd(1)-O(7)	86.24(6)
N(6A)-Cd(1)-O(7)	84.17(6)		

Symmetry codes: A -x + 1, -y + 1, -z + 1 for 1; A x, y, z - 1 for 2.

frequency $v_s(COO)$. The 1605 and 1454 cm⁻¹ absorptions for 1 are assigned to monodentate mode of COO because the calculated Δ value is 151 cm⁻¹, which is compared with that 144 cm^{-1} of the free CH₃COO⁻ [30]. The 1505 and 1397 cm^{-1} absorptions are assigned to bisdentate mode of COO because the calculated Δ value is 108 cm⁻¹, which is obviously smaller than 144 cm^{-1} of the free CH₃COO⁻. The 1598 and 1443 cm⁻¹ absorptions (the calculated Δ value is 155 cm^{-1}) are attributed to the monodentate COO, and 1505 and 1389 cm⁻¹ absorptions (the calculated Δ value is 116 cm⁻¹) are assigned to bisdentate COO for **2**. **1** and 2 both contain monodentate and bisdentate COO coordination modes which is confirmed by X-ray diffraction analysis. The triazole ring vibrations appear at 1536 and 1273 cm^{-1} for **1**, 1528 and 1281 cm⁻¹ for **2** [17,18]. The high frequency absorptions 3241 and 3141 cm⁻¹ for attributed to the O-H stretching vibrations of phba molecule for 1. The strong peaks from 3118 to 3386 cm^{-1} for 2 are assigned to the coordinated water molecule and OH group of phba molecule.

The crystal structure of 1 is made of neutral dimer metallacycle. In each dimer metallacycle, as shown in Fig. 1, two zinc(II) centers are connected by two btp molecules, resulting in a discrete $Zn_2(btp)_2$ 20-membered metallacycle which represents the smallest closed cyclic



Fig. 1. The dimeric structure of 1. Symmetry code: A -x + 1, -y + 1, -z + 1.

structure with a 1:1 metal-to-ligand ratio. Each zinc(II) center in **1** is in a fiver-coordinated distorted square-pyramidal geometry, coordinated by three oxygen atoms from two phba molecules $[Zn(1)-O(1) \ 2.153(2) \ \text{Å}; \ Zn(1)-O(2) \ 2.152(2) \ \text{Å}; \ Zn(1)-O(4) \ 1.9839(17) \ \text{Å}]$ and one nitrogen atom from one btp molecule $[Zn(1)-N(6A) \ 2.064(2) \ \text{Å}]$ in the plane positions, and one nitrogen atom from the other btp molecule $[Zn(1)-N(3) \ 2.066(2) \ \text{Å}]$ in the axial position.

Two phba molecules act as the different coordination modes. The COO group of one phba molecule acts as chelating coordination mode [O(1) and O(2)] to one zinc(II) atom, the COO group of the other phba molecule exhibits as monodentate coordination mode [O(4)]. The btp molecules show the *anti-gauche* conformation. The dihedral angle between two triazole rings is 64.0° . The Zn \cdots Zn separation *via* the bridging ligand btp in **1** is 10.116 Å, very closer to the metal-metal separation 10.447 Å in the reported btp ligand complex $[Co(btp)_2(NCS)_2]$ [19].

There are hydrogen bonding interactions between the hydrogen atoms of hydroxy groups of two phba molecules and the uncoordination N(2) atom of triazole ring and O(5) atom of the COO group of the adjacent dimers (Table 3 Fig. 2).

The crystal structure of **2** is made of one-dimensional zigzag chain (Fig. 3), which is different from the dimer structure of **1**. The coordination geometry of the Cd(II) atom in **2** is distorted octahedral; it is coordinated by two nitrogen atoms from the triazole rings of two symmetry-related btp ligands [Cd(1)–N(3) 2.2574(16) Å; Cd(1)–N(6A) 2.2572(16) Å] and two oxygen atoms from one COO group of phba molecule [Cd(1)–O(1) 2.4841(14) Å; Cd(1)–O(2) 2.3054(14) Å] in the equatorial plane, and two oxygen atoms from one phba molecule [Cd(1)–O(7) 2.4502(17) Å] in the axial positions.

Two phba molecules in 2 also act as the different coordination modes (the chelating and monodentate coordination modes) similar to that in 1. Each btp molecule in 2 coordinates to two Cd(II) atoms through its two triazole nitrogen atoms, thus acting as a bridging bidentate ligand to link two Cd(II) atoms and form a one-dimensional zigzag chain. The btp molecules also exhibit the *anti-gauche* conformation. The dihedral angle between two triazole rings is 21.6°, different from the dihedral angle 64.0° in 1. The Cd···Cd distance separated by btp bridge is

Table 3			
Hydrogen	bonding parameters (Å,	°) for	1

$D-H\cdots A \qquad \qquad d(D-H) \qquad d(H\cdots A) \qquad d(D\cdots A)$	(DHA)
$O(3)-H(3B)\cdots N(2)^{i}$ 0.84 2.00 2.797(3)	159.0
$O(6)-H(6C)\cdots O(5)^{ii}$ 0.84 1.78 2.606(3)	167.8
$O(7)-H(2W)\cdots N(2)$ 0.72(3) 2.26(3) 2.941(3)	160(3)
$O(7)-H(1W)\cdots O(1)$ 0.77(3) 2.01(3) 2.770(2)	167(3)

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



Fig. 2. The cell packing of 1.



Fig. 3. Viewing the one-dimensional zigzag chain of 2. Symmetry codes: A x, y, z - 1; B x, y, z - 2.

11.214 Å, equal to the *c* axis translation, which is small longer than the $Zn \cdot Zn$ distance (10.447 Å) in **1**.

It is interesting that the hydrogen bonding and π - π stacking interactions play an important role in the formation of crystal structure. There are intramolecular hydrogen bonding interactions between the hydrogen atoms of

hydroxy groups and the coordination water molecules, and the carboxylato-oxygen atoms of coordinated phba and the nitrogen atom of triazole ring [Table 4]. The C8–C13 phenyl ring and his symmetry-related C8–C13 (1 - x, 1 - y, -z) phenyl ring are absolutely parallel with the centeriod-to-centeriod distance 4.138 Å and plane

Table 4 Hydrogen bonding parameters (Å, °) for **2**

d(D-H)	$d(H \cdot \cdot \cdot A)$	$d(\mathbf{D} \cdot \cdot \cdot \mathbf{A})$	((DHA)
0.84	1.83	2.668(2)	179.6
0.84	1.83	2.665	171.7
0.72(3)	2.26(3)	2.941(3)	160(3)
0.77(3)	2.01(3)	2.770(2)	167(3)
	d(D–H) 0.84 0.84 0.72(3) 0.77(3)	$\begin{array}{ccc} d(D-H) & d(H\cdots A) \\ \hline 0.84 & 1.83 \\ 0.84 & 1.83 \\ 0.72(3) & 2.26(3) \\ 0.77(3) & 2.01(3) \end{array}$	$\begin{array}{c cccc} d(\mathrm{D-H}) & d(\mathrm{H}\cdots\mathrm{A}) & d(\mathrm{D}\cdots\mathrm{A}) \\ \hline 0.84 & 1.83 & 2.668(2) \\ 0.84 & 1.83 & 2.665 \\ 0.72(3) & 2.26(3) & 2.941(3) \\ 0.77(3) & 2.01(3) & 2.770(2) \end{array}$

Symmetry transformations used to generate equivalent atoms: a, -x + 1, -y + 1, -z; b, -x + 1, -y, -z + 1; c, -x + 2, -y + 1, -z + 1; d, -x + 2, -y + 1, -z.

distance 3.186 Å; the C15–C20 phenyl ring and his symmetry-related C15–C20 (1 - x, 1 - y, 1 - z) phenyl ring are absolutely parallel with the centeriod-to-centeriod distance 4.029 Å and plane distance 3.446 Å; the C1/C2/N1-N3 triazole ring and his symmetry-related C1/C2/N1/N3 (2 - x, 1 - y, 1 - z) triazole ring are absolutely parallel with the centeriod-to-centeriod distance 4.252 Å and plane distance 3.011 Å, exhibiting obvious π – π stacking interactions interphenyl rings and inter-triazole rings. The cell packing of **2** is shown in Fig. 4.

Previously Albada and coworkers reported two copper(II) complexes { $[Cu(btp)_2(CH_3CN)(H_2O)]$ (CF₃SO₃)₂}_n and { $[Cu(btp)_2(CH_3CN)_2]$ (CIO 4)₂}_n with two-dimensional network [18], and Zhao and coworkers synthesized a cobalt(II) complex [Co(btp)₂(NCS)₂]_n with one-dimensional double chain structure [19]. Compared the structure of **1** with **2**, the coordination geometry of the Cd(II) is six-coordination, in distorted octahedral, while the coordination environment of Zn(II) is a five-coordinated distorted square-pyramidal geometry, because the water molecule is coordinated to Cd(II) atom but there is no water in **1**. Zn(II) complex **1** is the neutral dimer Zn₂(btp)₂ metallacycle, while Cd(II) complex **2** forms the one-dimensional zigzag chain structure through the btp bridges.

Measurement of the solid state luminescence spectra of 1 and 2 reveals a strong blue emission maximum at approx-



Fig. 5. Solid state emission and excitation spectra of $\mathbf{1}$ and $\mathbf{2}$ at room temperature.

imately 402 and 394 nm, respectively, upon excitation at 340 nm (Fig. 5). This emission is neither metal-to-metal charge transfer (MLCT) nor ligang-to-metal transfer (LMCT) in nature since the Zn(II) and Cd(II) are difficult to oxidize or to reduce to its d¹⁰ configuration. Comparably, free phba and btp molecule in the solid state displays the emission band centered at about 395 and 410 nm, respectively, when excited at 340 nm. The luminescence emissions of **1** and **2** might therefore probably be assigned to the intraligand (π - π *) emission (combination of π - π * transition of phba and btp) [31,32].

On the basis of this work, further syntheses and structural studies of novel coordination polymers with flexible bis(triazole) ligands are also under way in our laboratory.

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Fig. 4. The cell packing of 2.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2006.10.028.

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