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Two complexes based on 1*H*-1,2,3-triazole-4,5-dicarboxylic acid: hydrothermal synthesis, crystal structures and spectral properties

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[Zn₃(tda)₂(bipy)₂(H₂O)₂·4H₂O]_{*n*} (**1**) and [Co₂(Htda)₂(H₂O)₆·5H₂O] (**2**) have been synthesized and characterized structurally by X-ray diffraction, where H₃tda = 1*H*-1,2,3-triazole-4,5-dicarboxylic acid and 2,2'-bipy = 2,2'-bipyridine. Their solid-state structures have been characterized by elemental analysis and IR spectroscopy. The molecular unit of **1** consists of two crystallographically unique Zn(II) ions assuming different coordination geometries, the tda³⁻ exhibits a hexadentate binding mode chelating three Zn(II) ions; neighboring Zn–Zn distances through tda³⁻ bridges are 5.910(6), 5.888(5), and 6.279(3) Å, respectively. In **2**, two neighboring Co(II) ions are bridged by two Htda²⁻ ligands, forming a binuclear structure, with Co–Co distance of 4.091 Å and is further linked to generate a 3-D structure via hydrogen bonds. Fluorescent of **1** was investigated.

Keywords: Complex; Hydrothermal synthesis; Crystal structure; Fluorescent property

1. Introduction

There has been much interest in crystal engineering of metal-organic frameworks (MOFs) in view of their superior functional properties and variety of potential applications [1, 2]. Much effort has been devoted to modify the building blocks and to control the assembled motifs for required products via selecting different organic ligands [3, 4]. Among them, particular attention has been paid to some heterocyclic dicarboxylic acids because the heteroatoms in these acids may serve as potential coordinating sites, resulting in the formation of structures of higher dimensions under hydrothermal conditions [5–8]. 1*H*-1,2,3-triazole-4,5-dicarboxylic acid (H₃tda) is one of such ligands with rigid planar multiple coordination sites, and the three, in which nitrogen atoms may work as useful coordination sites, and were useful in the construction of coordination polymers. On the other hand, H₃tda can be partially or fully deprotonated allowing the generation of coordination polymers with different structures, interesting topologies and properties. Considering that this ligand with various metals have produced intriguing poly-dimensional structures with complicated

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topologies, including examples of Rh(II), Cu(II), Mn(II), Cu(II)/Fe(II), Gd(III), and Cd(II) complexes [9–14], herein we present the hydrothermal synthesis, structure characterization, and spectral properties of $[\text{Zn}_3(\text{tda})_2(2,2'\text{-bipy})_2(\text{H}_2\text{O})_2 \cdot 4\text{H}_2\text{O}]_n$ (**1**) and $[\text{Co}_2(\text{Htda})_2(\text{H}_2\text{O})_6 \cdot 5\text{H}_2\text{O}]$ (**2**) ($\text{H}_3\text{tda} = 1\text{H}$ -1,2,3-triazole-4,5-dicarboxylic acid, 2,2'-bipy = 2,2'-bipyridine).

2. Experimental

2.1. Physical measurements

Infrared-spectra were recorded on a Nicolet-460 spectrophotometer using KBr discs. Thermal stability (TG-DTA) studies were carried out on a Dupont thermal analyzer from room temperature to 800°C at a rate of 10°C min⁻¹. Elemental analyses were performed with a PE-2400II apparatus. Emission spectra were taken on an F-4500 spectrofluorometer.

2.2. Materials and synthesis

All reagents and solvents employed were commercially available and used as received. H_3tda was synthesized according to the literature method [15]. As very little of **2** has been obtained, its thermogravimetric analysis and fluorescence have not been done.

2.2.1. Synthesis of $[\text{Zn}_3(\text{tda})_2(2,2'\text{-bipy})_2(\text{H}_2\text{O})_2 \cdot 4\text{H}_2\text{O}]_n$ (1**).** A mixture of $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.110 g, 0.5 mmol), H_3tda (0.078 g, 0.5 mmol) and 2,2'-bipy (0.082 g, 0.5 mmol) were dissolved in water (12 mL) and ethanol (3 mL), the pH of the solution was adjusted to be about 6–7 with 0.2 mol·L⁻¹ aqueous NaOH, then the mixed solution was stirred for 30 min at room temperature, transferred to and sealed in a 25 mL Teflon-lined stainless steel reactor, and then heated at 150°C for 72 h. Upon cooling to room temperature, colorless crystals were obtained. Yield: 56% based on Zn. Anal. Calcd for $\text{C}_{28}\text{H}_{28}\text{Zn}_3\text{N}_{10}\text{O}_{14}$: C, 36.37; H, 3.05; N, 15.14. Found: C, 36.32; H, 3.01; N, 15.18. IR (KBr, cm⁻¹): $\nu(\text{H-O-H})$, 3395; $\nu_{\text{as}}(\text{COO})$, 1593, 1562; $\nu_{\text{s}}(\text{COO})$, 1464.

2.2.2. Synthesis of $[\text{Co}_2(\text{Htda})_2(\text{H}_2\text{O})_6 \cdot 5\text{H}_2\text{O}]$ (2**).** The hydrothermal procedure for the synthesis of **2** is similar to that for **1** except for the replacement of $\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ with $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ (0.119 g, 0.5 mmol) and 2,2'-bipy was not added. Then the red crystals were obtained. Anal. Calcd for $\text{C}_8\text{H}_{24}\text{Co}_2\text{N}_6\text{O}_{19}$: C, 15.33; H, 3.83; N, 13.41. Found: C, 15.27; H, 3.88; N, 13.36. IR (KBr, cm⁻¹): $\nu(\text{H-O-H})$, 3386; $\nu_{\text{as}}(\text{COO})$, 1590, 1576; $\nu_{\text{s}}(\text{COO})$, 1432.

2.3. Crystallographic data collection and structure determination

The crystal structures were solved by direct methods using SHELXS-97 [16] and refinement on F^2 was performed using SHELXL-97 by full-matrix least-squares with anisotropic thermal parameters for all non-hydrogen atoms [17]. All hydrogens were

assigned common isotropic displacement factors and included in the final refinement by using geometrical restraints. Two single crystals of dimensions ca $0.20 \times 0.18 \times 0.15 \text{ mm}^3$ and $0.18 \times 0.16 \times 0.15 \text{ mm}^3$ were mounted on a Bruker APEX-II area-detector with graphite-monochromated Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$). Diffraction data were collected using the φ - ω scan mode at room temperature. Unit cell parameters were determined from $1.87^\circ \leq \theta \leq 25.05^\circ$ for **1** and $1.21^\circ \leq \theta \leq 25.05^\circ$ for **2**. Experimental details for the structural determination of **1** and **2** are presented in tables 1 and 2.

3. Results and discussion

3.1. Crystal structure of **1**

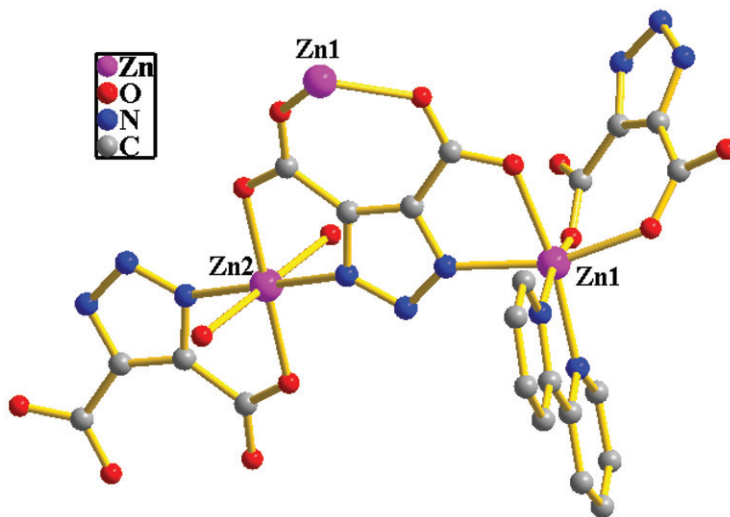
The crystal structure of **1** is illustrated in figure 1, with two crystallographically unique Zn(II) ions (Zn1 and Zn2) assuming different coordination geometries in an asymmetric unit. Zn1 is six-coordinate, coordinated with two nitrogens from bipy and three oxygens and one nitrogen from two tda³⁻ ligands in a distorted octahedron. The Zn1–N and Zn1–O bond distances range from 2.1313(19) to 2.1594(18) \AA and 2.0758(16) to 2.1751(15) \AA , respectively. The *cis*-angles at Zn1 fall in the range of 75.23(6)–168.75(7) $^\circ$. Zn2 is also six-coordinate but coordinated with two oxygen atoms from two water molecules, two nitrogen atoms, and two oxygen atoms from two tda³⁻ ligands. The Zn2–N and bond distances are 2.0873(18) \AA and the Zn2–O bond distances are

Table 1. Crystal data and structure refinement for **1**.

Formula	C ₂₈ H ₂₈ Zn ₃ N ₁₀ O ₁₄
Formula weight	924.71
Temperature (K)	273(2)
Wavelength (\AA)	0.71073
Crystal system	Monoclinic
Space group	<i>P</i> 2(1)/ <i>n</i>
Unit cell dimensions (\AA , $^\circ$)	
<i>a</i>	12.4208(13)
<i>b</i>	7.6914(8)
<i>c</i>	18.812(2)
β	97.015(2)
Volume (\AA^3), <i>Z</i>	1783.7(3), 2
Calculated density (g cm^{-3})	1.722
Absorption coefficient (mm^{-1})	2.082
<i>F</i> (000)	936
θ range for the data collection ($^\circ$)	1.877–25.05
Limiting indices	$-14 \leq h \leq 7$, $-9 \leq k \leq 9$, $-22 \leq l \leq 21$
Reflections collected/unique	9037/3160 [<i>R</i> (int) = 0.0205]
Refinement method	Full-matrix least-squares on <i>F</i> ²
Absorption correction	None
Data/restraints/parameters	3160/9/250
Goodness-of-fit on <i>F</i> ²	0.991
Final <i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> ₁ = 0.0235, <i>wR</i> ₂ = 0.0502
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0283, <i>wR</i> ₂ = 0.0608
Largest difference peak and hole ($e \text{\AA}^{-3}$)	0.353 and -0.304

Table 2. Crystal data and structure refinement for **2**.

Formula	C ₈ H ₂₄ Co ₂ N ₆ O ₁₉
Formula weight	626.18
Temperature (K)	293(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions (Å, °)	
<i>a</i>	34.827(11)
<i>b</i>	7.0171(14)
<i>c</i>	17.801(4)
β	104.80(3)
Volume (Å ³), <i>Z</i>	4206.0(18), 8
Calculated density (g cm ⁻³)	1.921
Absorption coefficient (mm ⁻¹)	1.679
<i>F</i> (000)	2480
θ range for the data collection (°)	1.21–25.05
Limiting indices	−41 ≤ <i>h</i> ≤ 41, −8 ≤ <i>k</i> ≤ 8, −20 ≤ <i>l</i> ≤ 21
Reflections collected/unique	3714/3714 [<i>R</i> (int) = 0.0000]
Refinement method	Full-matrix least-squares on <i>F</i> ²
Absorption correction	None
Data/restraints/parameters	3714/0/310
Goodness-of-fit on <i>F</i> ²	1.041
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> ₁ = 0.0329, <i>wR</i> ₂ = 0.0953
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0400, <i>wR</i> ₂ = 0.1028
Largest difference peak and hole (e Å ⁻³)	0.470 and −0.416

Figure 1. The coordination environment of Zn^{II} and tda³⁻ in **1**.

2.1384(16) and 2.1763(19) Å, respectively. The *cis*-angles at Zn2 fall in the range of 78.12(6)–180.000(1)° in a slightly distorted octahedron. The tda³⁻ is hexadentate (figure 1), chelating two Zn1 and one Zn2 ions. Neighboring Zn–Zn distances through tda³⁻ bridges are 5.910(6), 5.888(5), and 6.279(3)Å, respectively. As shown in figure 2,

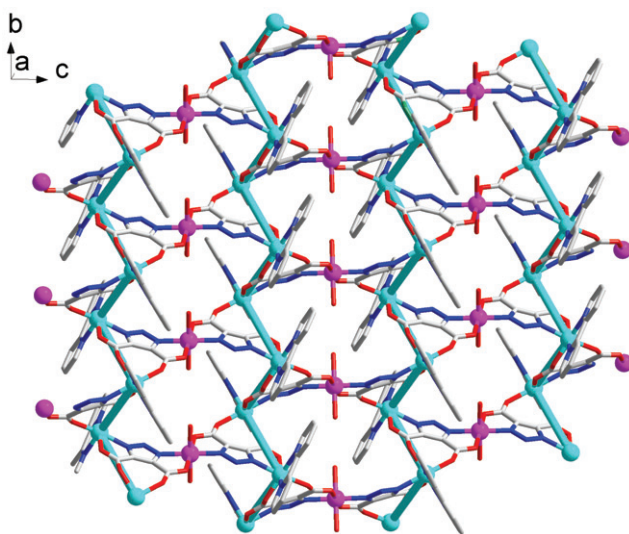


Figure 2. The 2-D infinite layer framework of **1**.

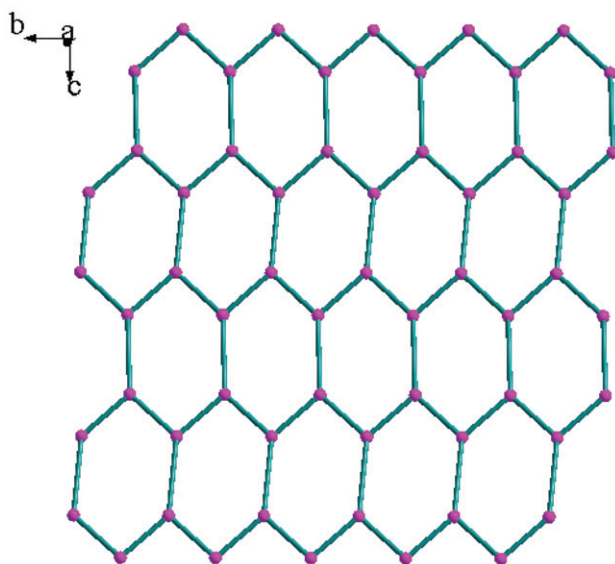
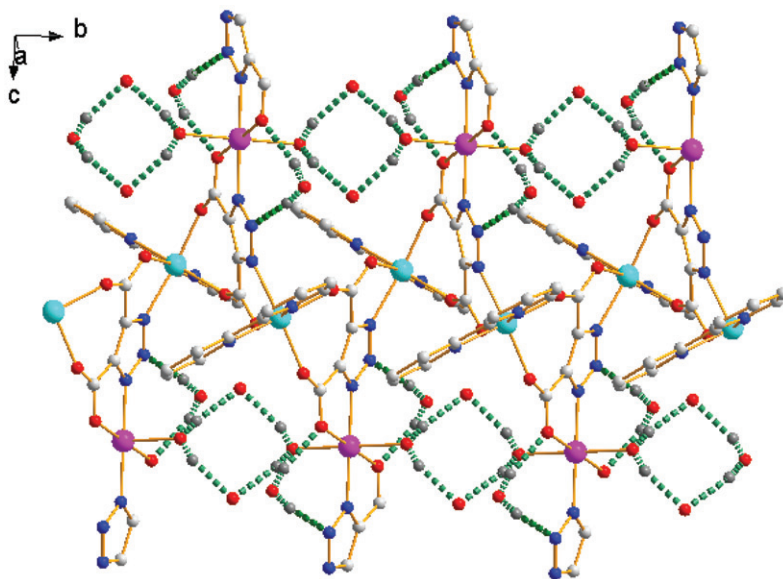


Figure 3. The (6^3) topological net of **1**.

each tda^{3-} further bridges two crystallographically identical Zn1 ions to form a 1-D helical/zigzag chain in the b direction. These chains are linked through Zn2 coordinated to tda^{3-} in the c direction to form a 2-D infinite layer. Since tda^{3-} connects three metal ions, it can be rationalized to be (6^3) topological net with tda^{3-} acting as a three node (figure 3).

There are three symmetry-independent intermolecular hydrogen bonds in the crystal structure of **1**. Two intermolecular hydrogen bonds are formed between crystal-lattice

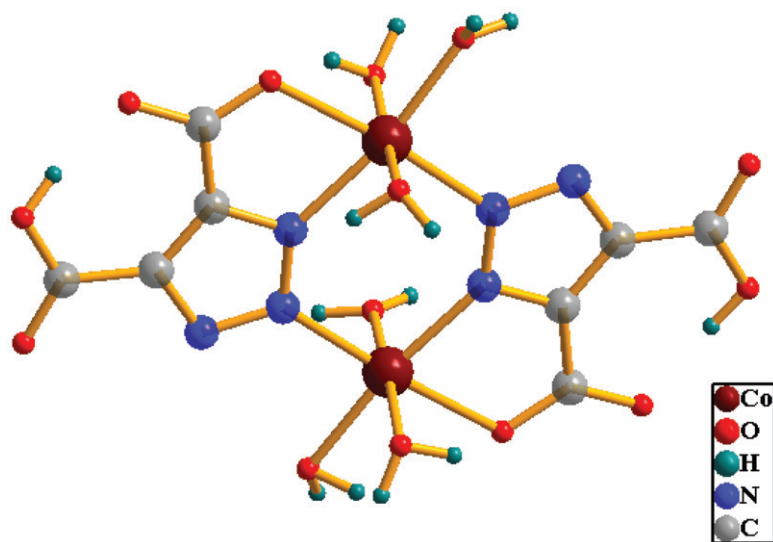
Figure 4. The hydrogen bonds of **1**.

water and coordinated water: O(5)–H(15)⋯O(7)^{*i*} (O⋯O 2.748 Å, H⋯O 1.935 Å, angle 161.58°, symmetry code ^{*i*}: $-x+1, -y-1, -z$), O(5)–H(16)⋯O(7)^{*ii*} (O⋯O 2.836 Å, H⋯O 2.010 Å, angle 167.48°, symmetry code ^{*ii*}: $+x, +y+1, +z$). Hydrogen bonds between coordinated water and the crystal-lattice water form a tetrameric core (figure 4). The other kind of intermolecular hydrogen bond is between the oxygen atom from coordinated water and oxygen atom of tda³⁻: O(6)–H(18)⋯O(4)^{*iii*} (O⋯O 2.830 Å, H⋯O 2.011 Å, angle 160.47°, symmetry code ^{*iii*}: $-x+1, -y-1, -z$). There also exists intramolecular hydrogen bond O(6)–H(17)⋯N(2)^{*iv*} (O⋯N 2.799 Å, H⋯N 2.011 Å, angle 152.83°, symmetry code ^{*iv*}: $+x, +y, +z$), as shown in figure 4. These hydrogen bonds play an important role in stabilization of the layer structure.

3.2. Crystal structure of **2**

X-ray crystallography shows that **2** is binuclear constructed by Htda²⁻ and Co(II) associated with water molecules, and crystallizes in space group *C2/c*. The asymmetric unit consists of two Co(II) ions, two Htda²⁻, and eleven water molecules in which six of them are coordinated to Co(II). As shown in figure 5, Co(1) and Co(2) are both six-coordinate [CoN₂O₄] and are coordinated by four oxygen atoms, two from two Htda²⁻ and two from water molecules with distorted octahedral geometries. Co–O bond lengths are in the range of 2.090(2)–2.172(3) Å and 2.043(3)–2.165(3) Å for Co(1) and Co(2), respectively. The Co–N bond lengths are 2.107(3) and 2.108(3) Å for Co(1), while 2.085(3) and 2.094(3) Å for Co(2). The bond angles are in the range 75.4(3)–168.6(4)° and 78.7(3)–168.9(4)° for Co(1) and Co(2), respectively. The Htda²⁻ links two Co(II) ions through tridentate coordination mode (figure 5).

There exist 21 symmetry-independent hydrogen bonds in the crystal structure among coordinated water and uncoordinated water molecules as well as nitrogen atoms

Figure 5. The coordination environments of Co(II) and Htda²⁻ in **2**.Table 3. Hydrogen bonds in **2**.

D–H...A	<i>d</i> (D–H)	<i>d</i> (H...A)	<i>d</i> (D...A)	∠DHA	Symmetry code
O(17)–H(25)...O(1)	0.8501	2.003	2.845	170.64	+ <i>x</i> , + <i>y</i> , + <i>z</i>
O(19)–H(28)...O(2)	0.8500	2.025	2.871	173.37	+ <i>x</i> , + <i>y</i> , + <i>z</i>
O(14)–H(19)...O(17)	0.8500	1.990	2.834	171.87	+ <i>x</i> , – <i>y</i> , –1/2+ <i>z</i>
O(19)–H(29)...O(13)	0.8500	1.971	2.816	172.64	– <i>x</i> , – <i>y</i> , – <i>z</i>
O(13)–H(17)...O(10)	0.8501	2.135	2.968	163.32	+ <i>x</i> , – <i>y</i> , –1/2+ <i>z</i>
O(12)–H(14)...O(17)	0.8501	1.838	2.645	157.92	+ <i>x</i> , – <i>y</i> +1, –1/2+ <i>z</i>
O(12)–H(15)...O(8)	0.8499	1.879	2.709	165.27	– <i>x</i> +1/2, – <i>y</i> +1/2, – <i>z</i>
O(10)–H(11)...O(4)	0.8500	2.063	2.668	127.44	– <i>x</i> , – <i>y</i> , – <i>z</i>
O(18)–H(27)...N(3)	0.8500	2.395	3.056	135.12	+ <i>x</i> , – <i>y</i> +1, + <i>z</i> +1/2
O(15)–H(21)...N(6)	0.8499	2.139	2.955	160.94	– <i>x</i> +1/2, + <i>y</i> +1/2, – <i>z</i> +1/2
O(15)–H(20)...O(6)	0.8500	2.032	2.868	168.05	– <i>x</i> +1/2, – <i>y</i> +1/2, – <i>z</i>
O(11)–H(13)...O(5)	0.8500	1.916	2.761	172.92	+ <i>x</i> , – <i>y</i> +1, + <i>z</i> –1/2
O(11)–H(12)...O(15)	0.8502	2.040	2.885	172.74	+ <i>x</i> , + <i>y</i> , + <i>z</i>
O(9)–H(9)...O(16)	0.8499	1.963	2.773	159.04	+ <i>x</i> , + <i>y</i> , + <i>z</i>
O(10)–H(10)...O(16)	0.8500	2.004	2.673	134.94	+ <i>x</i> , + <i>y</i> –1, + <i>z</i>
O(16)–H(23)...O(15)	0.8501	1.915	2.759	171.75	– <i>x</i> +1/2, + <i>y</i> +1/2, – <i>z</i> +1/2
O(16)–H(23)...O(11)	0.8501	2.431	3.075	132.99	+ <i>x</i> , + <i>y</i> , + <i>z</i>
O(16)–H(22)...O(8)	0.8500	2.351	3.008	134.34	– <i>x</i> +1/2, + <i>y</i> +1/2, – <i>z</i> +1/2
O(18)–H(26)...O(17)	0.8498	2.075	2.797	142.37	+ <i>x</i> , + <i>y</i> , + <i>z</i>
O(17)–H(24)...O(18)	0.8500	2.067	2.797	143.40	+ <i>x</i> , + <i>y</i> , + <i>z</i>
O(18)–H(27)...O(19)	0.8500	2.522	3.190	136.15	+ <i>x</i> , + <i>y</i> +1, + <i>z</i>

and oxygen atoms of Htda²⁻ (table 3). These hydrogen bonds link the whole binuclear structure into a 3-D supermolecular structure, as shown in figure 6. A rarely reported 2D water layer (figure 7) can be obtained between coordinated water and uncoordinated water in a 48-membered water ring consisting of 24 oxygen atoms and 24 hydrogen atoms (figure 8). These hydrogen bonds play an important role in stabilization of the 3D structure of **2**.

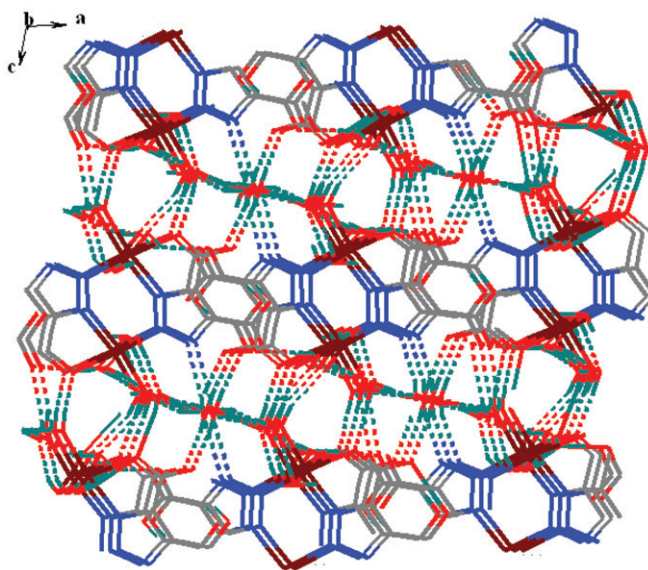


Figure 6. The 3-D structure of **2** connected by hydrogen bonds.

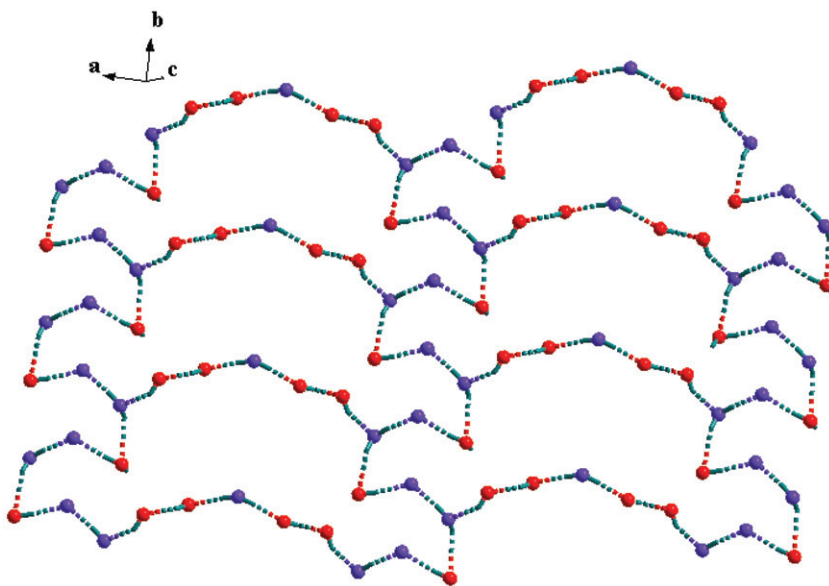


Figure 7. The 2-D water layer structure connected by hydrogen bonds only among water of **2**.

3.3. Summary of the structures

The H₃tda ligand (1*H*-1,2,3-triazole-4,5-dicarboxylic acid) can be partially or fully deprotonated acting in different coordination modes [9, 13, 18] when coordinating to

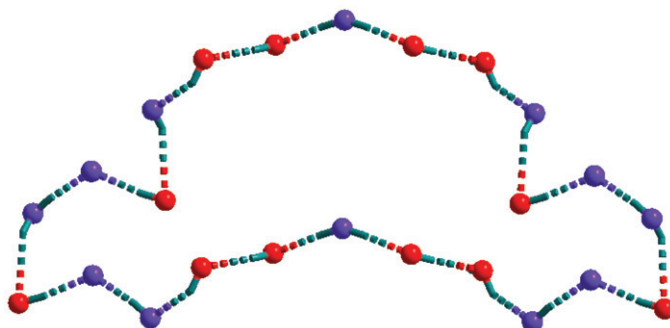


Figure 8. The 48-member water ring connected by hydrogen bonds between the coordinated water (red) and uncoordinated water (purple) of **2**.

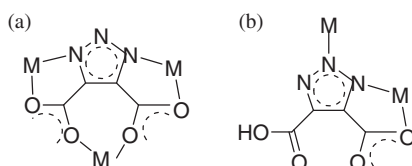


Figure 9. (a) The coordination mode of tda^{3-} in **1**; (b) the coordination mode of Htda^{2-} in **2**.

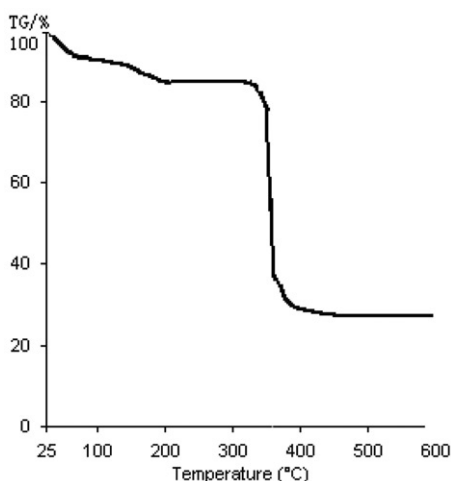
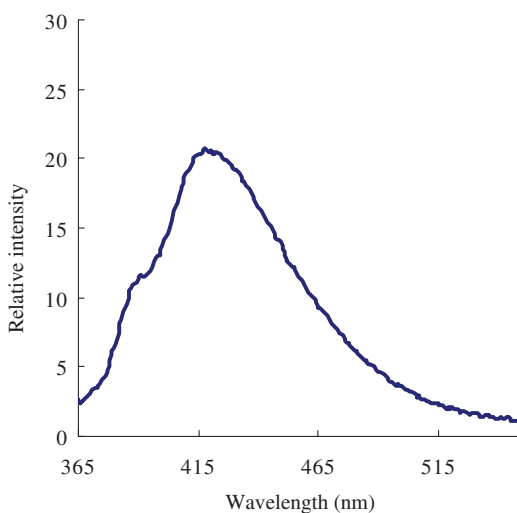
metal ions under different conditions; two coordination modes have been observed in **1** and **2** (figure 9). In **1**, H_3tda is fully deprotonated to tda^{3-} and adopt hexadentate coordination mode chelating three $\text{Zn}(\text{II})$ ions, while in **2** Htda^{2-} adopt tridentate coordination mode connecting two $\text{Co}(\text{II})$ ions. Because of different coordination modes of tda^{3-} and Htda^{2-} in **1** and **2**, different structures are displayed.

3.4. Thermogravimetric analysis (TGA) of **1**

Thermogravimetric analysis (TGA) for **1** was performed under a flow of N_2 . The weight loss of 10.64% during the first and second steps from 30°C to 208°C corresponds to the loss of four crystal-lattice water molecules and two coordinated water molecules (Calcd. 11.69%). The third-step weight loss of 61.68% (calcd 61.91%) from 320°C to 404°C corresponds to the decomposition of tda^{3-} and bipy (figure 10). Assuming the residue is ZnO , the observed weight (27.68%) is in good agreement with the calculated value (26.41%).

3.5. Luminescence of **1**

Luminescent complexes are of interest because of their various applications in chemical sensor, photochemistry, and structure electroluminescence displays [19, 20]. The synthesis of metal-organic complexes by judicious choice of conjugated organic spacers and transition metal centers can be an efficient method for obtaining new luminescent

Figure 10. The TGA of **1**.Figure 11. View of emission spectra of **1** in the solid state at room temperature.

materials, especially for d^{10} systems [21–24]. The solid-state luminescence spectrum of **1** has been recorded in the solid state at room temperature. Complex **1** exhibits strong fluorescence with an emission maximum at 422 nm upon excitation at 362 nm (figure 11). The emission is not metal-to-ligand charge transfer (MLCT) or ligand-to-metal charge transfer (LMCT) because Zn(II) is difficult to oxidize or reduce. The d^{10} ion is electrochemically stable, and thus, when coordinated to ligands, is not significantly affected by ligand-field-effect [25, 26]. Therefore, the emissions of **1** are assigned to ligand-based $\pi^*-\pi$ and $n^*-\pi$ transitions rather than ligand-to-metal charge transfer (LMCT) [27–29].

Supplementary material

CCDC 821425 (1) and 829404 (2) contain supplementary Crystallographic data (excluding structure factors) for this article. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre (CCDC) (12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336033).

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