

LETTER

A Novel 3D Cd^{II}-CP Based on a Bi-functional (N-/O-Sites) Triazole-modified Carboxyl Ligand: Structure, Topology and Photoluminescence^①

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ABSTRACT With triazole-modified derivative as link, one new framework [Cd₅(μ₅-HL²⁻)₂(μ₄-L³⁻)₂(H₂O)₁₀] 2H₂O (**1**, H₃L = 2-(1,2,4-triazol-1-yl)benzene-1,3,5-tricarboxylic acid) has been hydrothermally synthesized and characterized by single-crystal X-ray diffraction, powder X-ray diffraction and elemental analysis. Complex **1** exhibits a 3D (3,4,6)-connected net cross-linked by binuclear [Cd₂(COO)₅]⁻ cluster and mononuclear Cd(II) geometry via HL²⁻/L³⁻ ligands. It crystallizes in triclinic system, space group *P* $\bar{1}$ with *a* = 7.7411(11), *b* = 9.8813(15), *c* = 20.177(3) Å, *α* = 85.547(4)°, *β* = 80.603(4)°, *γ* = 67.976(3)°, *V* = 1411.3(4) Å³, *Z* = 1, *M_r* = 1876.89 g/mol, *D_c* = 2.208 mg/m³, *μ* = 1.97 mm⁻¹, *F*(000) = 918, *GOOF* = 1.03, the final *R* = 0.0869 and *wR* = 0.1950 for 3127 observed reflections with *I* > 2σ(*I*). The solid photoluminescent behaviour of **1** has been further functionally investigated.

Keywords: triazole, Cd(II) complex, crystal structure, fluorescence property;

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1 INTRODUCTION

The structural diversity, controllability and post-modification of coordination polymers (CPs) as well as their potential applications in various fields (such as gas storage and separation^[1-3], optics^[4,5], catalysis^[6], and magnetochemistry^[7]) still receive extensive attention. Fluorescent CPs are treated as an emerging optical material, exhibiting high application value in the fields such as photochemical sensors and electro-luminescent displays^[8,9].

The formation of most coordination polymers (CPs) is self-assembly driven by coordination^[10]. Many factors affect the assembly process, such as organic link, metal ion, temperature, pH, solvent and other weak interactions. Unfortunately up to now, the prediction and control for the final product are still a big challenge. During the self-assembly process of CPs, the ligand as an important component plays a direct impact on the final structure. A variety of typical bi-topic ligands, including pyridine carboxylate^[11], amino polycarboxylic acid^[12] and N-containing aromatic carboxylic acid^[13], represent attractive constituents for cross-linked building blocks. 1,2,4-Triazole is a five-membered ring

molecule containing three N atoms, and each N has a pair of donor electrons. By using substituents to modify the triazole, a variety of derivative ligands can be obtained. Triazole-modified aromatic carboxylic acid, as a bi-functional ligand containing multiple coordination sites (N/O), has been proved particularly successful^[14-16]. Some obvious reasons for this success are briefly mentioned: (a) Aromatic and triazole rings can produce intramolecular conjugated π bonds; (b) One or more carboxyl groups in the ligand tend to provide diverse coordination modes; (c) Neutral N atoms can easily coordinate with metal ions; (d) The different Pearson hardness^[17] of N/O coordination sites favors cross-linking of different cations and conveys additional stability to metal coordination.

Given the above-mentioned background, we use 2-(1,2,4-triazol-1-yl)benzene-1,3,5-tricarboxylic acid (H₃L) as ligand and Cd(II) ions as coordination centers to construct a new 3D framework. Here, we report the synthesis, characterization and luminescence property of complex [Cd₅(μ₅-HL²⁻)₂(μ₄-L³⁻)₂(H₂O)₁₀] 2H₂O (**1**). The simplified grid of complex **1** is described into a (3,4,6)-*conn* topological net.

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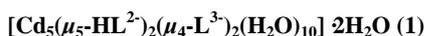
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2 EXPERIMENTAL

2.1 Materials and methods

All the reagents used in this work were commercially available. Elemental analyses for C, H, and N components were determined on a Perkin-Elmer analyzer. Powder X-ray diffraction patterns were collected on a D/Max-2500 X-ray diffractometer using Cu-K α radiation. Solid fluorescent spectrum was measured on a Varian Cary Eclipse Fluorescence spectrophotometer.

2.2 Synthesis of complex



A mixture of CdCl₂·H₂O (0.102 g, 0.33 mmol), H₃L (0.062 g, 0.25 mmol) and H₂O/CH₃CN (4 mL, 1:3 v/v) was placed into a 25 mL PTFE vessel sealed with a stainless-steel reactor and heated to 120 °C for 3 days. Colorless crystals with a yield of 65.2% were obtained through the cooling process (2 °C/h). Elemental analysis found/calcd. (%) for **1**: C, 29.46/29.54; H, 2.14/2.24; N, 9.03/8.96.

2.3 X-ray crystallography

Single-crystal X-ray diffraction data for complex **1** were collected on a Bruker APEX-II CCD diffractometer. Specific X-ray source and collection temperature are limited to Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$) at 296(2) K. Unit cell parameters were determined by SMART program. Absorption was corrected by the multi-scan method using SADABS program^[18]. Structure was solved *via* direct methods employed in the SHELXS-2018 program^[19] and refined by full-matrix least-squares techniques against F^2 . Non-hydrogen atoms were assigned anisotropically. Hydrogen atoms (C–H) were defined geometrically and refined using a riding model approximation, while H atoms (O–H) were located from Fourier maps and refined as riding with O–H bond lengths of 0.82~0.84 Å ($U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$). One triazole ring shows conformational disorder. The PART instruction was used to split both conformers, and the sum of the occupancies was constrained to 1. Selected bond lengths, bond angles and H-bonds are shown in Tables 1 and 2.

Table 1. Selected Bond Lengths (Å) and Bond Angles (°)

Bond	Dist.	Bond	Dist.	Bond	Dist.
Cd(1)–N(3) ⁱ	2.271(9)	Cd(1)–O(5) ⁱⁱ	2.291(9)	Cd(1)–O(7)(H ₂ O)	2.322(10)
Cd(1)–O(14)	2.350(9)	Cd(1)–O(1)	2.352(7)	Cd(1)–O(2)	2.427(8)
Cd(2)–O(15) ⁱⁱⁱ	2.213(8)	Cd(2)–O(8)(H ₂ O)	2.242(10)	Cd(2)–O(9)(H ₂ O)	2.291(9)
Cd(2)–O(11) ^{iv}	2.299(8)	Cd(2)–O(6) ⁱⁱ	2.312(8)	Cd(2)–O(1)	2.379(8)
Cd(3)–N(6) ^v	2.245(9)	Cd(3)–N(6)	2.245(9)	Cd(3)–O(17)	2.273(8)
Cd(3)–O(17) ^v	2.273(8)	Cd(3)–O(16)	2.294(8)	Cd(3)–O(16) ^v	2.294(8)
Angle	(°)	Angle	(°)	Angle	(°)
N(3) ⁱ –Cd(1)–O(5) ⁱⁱ	95.4(3)	O(5) ⁱⁱ –Cd(1)–O(7)	92.7(4)	O(5) ⁱⁱ –Cd(1)–O(14)	82.3(3)
N(3) ⁱ –Cd(1)–O(7)	89.2(4)	N(3) ⁱ –Cd(1)–O(14)	84.7(3)	O(7)–Cd(1)–O(14)	171.7(3)
N(3) ⁱ –Cd(1)–O(1)	142.2(3)	O(7)–Cd(1)–O(1)	86.3(3)	N(3) ⁱ –Cd(1)–O(2)	89.2(3)
O(5) ⁱⁱ –Cd(1)–O(1)	122.2(3)	O(14)–Cd(1)–O(1)	101.9(3)	O(5) ⁱⁱ –Cd(1)–O(2)	173.9(3)
O(7)–Cd(1)–O(2)	91.4(3)	O(1)–Cd(1)–O(2)	53.5(2)	O(15) ⁱⁱⁱ –Cd(2)–O(9)	87.0(3)
O(14)–Cd(1)–O(2)	94.2(3)	O(15) ⁱⁱⁱ –Cd(2)–O(8)	89.7(4)	O(8)–Cd(2)–O(9)	170.0(3)
O(15) ⁱⁱⁱ –Cd(2)–O(11) ^{iv}	99.0(3)	O(9)–Cd(2)–O(11) ^{iv}	105.3(3)	O(8)–Cd(2)–O(6) ⁱⁱ	85.8(4)
O(8)–Cd(2)–O(11) ^{iv}	84.6(3)	O(15) ⁱⁱⁱ –Cd(2)–O(6) ⁱⁱ	165.9(3)	O(9)–Cd(2)–O(6) ⁱⁱ	95.1(3)
O(11) ^{iv} –Cd(2)–O(6) ⁱⁱ	93.9(3)	O(8)–Cd(2)–O(1)	84.2(3)	O(11) ^{iv} –Cd(2)–O(1)	166.1(3)
O(15) ⁱⁱⁱ –Cd(2)–O(1)	89.1(3)	O(9)–Cd(2)–O(1)	86.3(3)	O(6) ⁱⁱ –Cd(2)–O(1)	77.2(3)
N(6) ^v –Cd(3)–N(6)	180.0	N(6)–Cd(3)–O(17)	94.6(3)	N(6)–Cd(3)–O(17) ^v	85.4(3)
N(6) ^v –Cd(3)–O(17)	85.4(3)	N(6) ^v –Cd(3)–O(17) ^v	94.6(3)	O(17)–Cd(3)–O(17) ^v	180.0
N(6) ^v –Cd(3)–O(16) ^v	89.6(3)	O(17)–Cd(3)–O(16) ^v	91.7(3)	N(6) ^v –Cd(3)–O(16)	90.4(3)
N(6)–Cd(3)–O(16) ^v	90.4(3)	O(17) ^v –Cd(3)–O(16) ^v	88.3(3)	N(6)–Cd(3)–O(16)	89.6(3)
O(17)–Cd(3)–O(16)	88.3(3)	O(17) ^v –Cd(3)–O(16)	91.7(3)	O(16) ^v –Cd(3)–O(16)	180.0

Symmetry codes: (i) $-x+1, -y+2, -z+1$; (ii) $x+1, y-1, z$; (iii) $x-1, y, z$; (iv) $x, y-1, z$; (v) $-x+3, -y+1, -z$

Table 2. Hydrogen Bond Lengths (Å) and Bond Angles (°)

D–H···A	d(D–H)	d(H···A)	d(D···A)	$\angle\text{DHA}$
O(18)···H(18B)···O(10) ^{iv}	0.82	2.15	2.826(17)	140
O(18)···H(18A)···N(2) ⁱⁱ	0.82	2.41	3.15(2)	149
O(17)···H(17B)···O(13) ^{viii}	0.82	2.22	2.664(11)	114

To be continued

O(17) ···H(17A) ···O(12) ^{vi}	0.82	1.87	2.666(11)	163
O(16) ···H(16B) ···O(11) ^{vi}	0.82	1.99	2.779(12)	162
O(16) ···H(16A) ···O(4) ^{ix}	0.82	1.99	2.769(12)	158
O(9) ···H(9B) ···O(5) ^{iv}	0.82	2.45	3.260(13)	169
O(8) ···H(8B) ···O(13)	0.82	2.46	2.889(13)	113
O(8) ···H(8A) ···N(5)	0.82	2.13	2.897(15)	156
O(7) ···H(7B) ···O(9)	0.82	2.12	2.830(13)	145
O(7) ···H(7A) ···O(18) ^x	0.82	1.80	2.603(17)	167
O(3) ···H(3) ···O(12) ^{ix}	0.82	1.79	2.593(10)	165

Symmetry codes: (ii) $x+1, y-1, z$; (iv) $x, y-1, z$; (vi) $x+2, y+2, z$; (viii) $x+1, y, z$; (ix) $-x+1, -y+2, -z$; (x) $-x+1, -y+1, -z+1$

3 RESULTS AND DISCUSSION

3.1 Structural description for 1

[Cd₅(μ₅-HL²⁻)₂(μ₄-L³⁻)₂(H₂O)₁₀] · 2H₂O (1). Complex 1 crystallizes in triclinic space group $P\bar{1}$, showing a 3D framework. The asymmetric unit consists of two and a half crystallographically independent Cd(II) ions, one μ₅-HL²⁻ anion, one μ₄-L³⁻ anion, five metal coordination water molecules and one lattice water molecule. As depicted in Fig. 1,

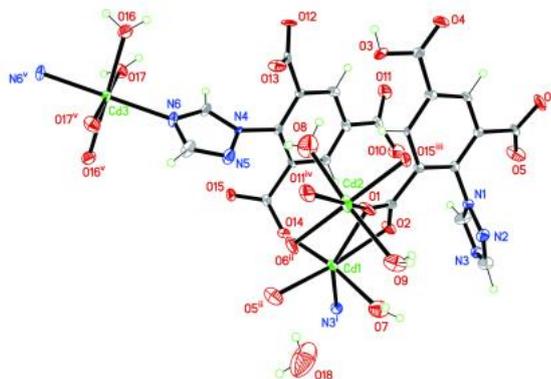


Fig. 1. ORTEP view of the coordination environments of Cd(II) ions in complex 1

Interestingly, three carboxylate groups of H₃L were deprotonated partially or completely to give two kinds of anions HL²⁻ and L³⁻ in the reaction system, resulting in HL²⁻ and L³⁻ anions which display specific μ₅-κN:κOO':κO':κO'':κO''' and μ₄-κN:κO:κO':κO'' coordination modes (Fig. 2). The dihedral angles formed by triazole and phenyl rings in HL²⁻ and L³⁻ ligands amount to 73.796(0.620)° and 63.085(0.333)° in order to reduce steric strain. H₃L as a typical bi-topic ligand possesses two slightly different Pearson hardness coordination sites (O and N). Negative carboxylate groups tend to bridge multiple metal centers to form cluster units. Neutral triazole nitrogen atom auxiliarily coordinated with above metals to expand the entire structure. As shown in Fig. 3a, atoms Cd(1) and Cd(2) are bonded to give a binuclear [Cd₂(COO)₅]⁻ secondary building

three distinct Cd(II) ions all adopt a distorted octahedral geometry. Cd(1) displays a seriously distorted octahedral geometry by coordinating as a [CdNO₅] mode. Cd(2) and Cd(3) are also coordinated with six atoms, which promotes the conformation of octahedral geometry. Besides, Cd(3) is located on the inversion center contributing to half occupancy. All of the Cd–O bond lengths fall in the range of 2.213(8)~2.427(9) Å, and the Cd–N lengths are 2.164(8)~2.245(9) Å.

unit (SBU) by carboxylate groups, with the short inter-SBU separation Cd(1) ···Cd(2) to be 3.8072(4) Å. Along the *ab* plane, binuclear SBU units are linked through benzene carboxylate moieties to form a 2D layer ignoring the participation of N sites. By a self-assembly process, adjacent layers are connected by triazole N coordination sites and *via* Cd(3) atoms (purple color) to develop into a 3D framework (Fig. 3b). In the simplified net, HL²⁻ connects three binuclear SBUs. Unlike HL²⁻, L³⁻ links three SBUs and one more extra Cd(3) atom to extend toward four directions. Considering the binuclear SBU as a 6-c node, HL²⁻ and L³⁻ ligands as 3-c and 4-c nodes, the whole structure can be simplified as a 3D (3,4,6)-*conn* topological network. The corresponding Schläfli symbol is determined to {4 6² 8³}₂{4³}₂{4⁴ 6⁸ 8³}₂ by topological analysis^[20] (Fig. 4).

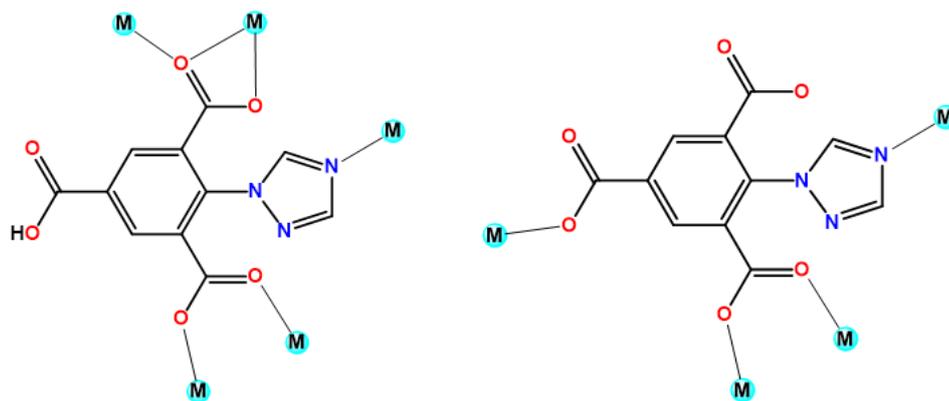
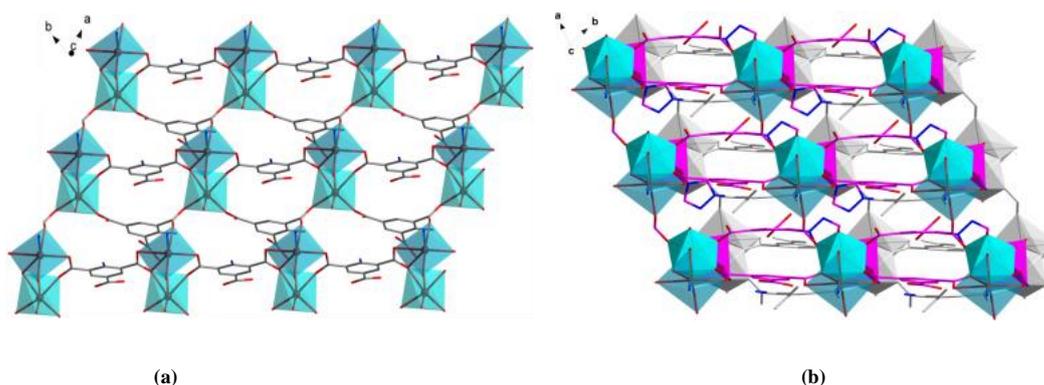
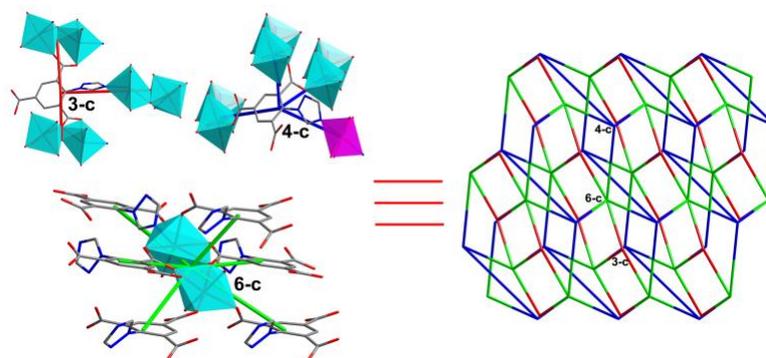
Fig. 2. Two kinds of coordination modes in H₃L

Fig. 3. (a) 2D network built from O-coordination sites, (b) View of the 3D framework connected by triazole N-coordination sites

Fig. 4. Topologically (3,4,6)-connected network assembled by 3-/4-/6-conn nodes in **1**

3.2 Powder X-ray diffraction (PXRD) analysis

PXRD experiment of **1** has been carried out to establish its crystalline phase purity. The experimental patterns of bulk samples are consistent with the simulated patterns corresponding to single crystal data (Fig. 5). Therefore, we can confirm the identity of the bulk material with single crystal structures.

3.3 Luminescent property

As an emerging fluorescent material, fluorescent CPs have the advantages of simple synthesis, controllable structure, high luminous efficiency, and pure chromaticity^[21-22]. As an important component, organic ligands usually contain large

π -conjugated systems. They can coordinate with metal ions containing rich d^{10} electrons (such as Cd(II) or Zn(II)) to form fluorescent CPs^[23]. The close packing of structure and the enhancement of system rigidity will induce the complex to generate excellent photoluminescence^[24, 25]. Thus, it has shown extremely high application values in the field of optical materials, such as photochemical sensors and electro-luminescent displays^[8, 9]. In this line of argument, fluorescence monitoring represents a decisive analytical tool for exploring the photoluminescence of fluorescent CPs. Solid state fluorescence of **1** has been investigated at room temperature. Emission spectrum of **1** shows a maximum

emission peak at 371 nm under the UV excitation of 285 nm (Fig. 6). Applying the CIE1931 program, the fluorescence chromaticity coordinates are calculated as (0.1521, 0.0257), which falls in blue light region (inset in Fig. 6). It is speculated that the reason for fluorescence may be the charge

transfer from ligand to ligand (LLCT) or intra-ligand ($n-\pi^*$ or $\pi-\pi^*$) emission in the complicated network. The metal-to-ligand MLCT or ligand-to-metal LMCT in Cd(II) complex can be completely ignored, because the full electron Cd(II) ion is difficult to oxidize or reduce^[26].

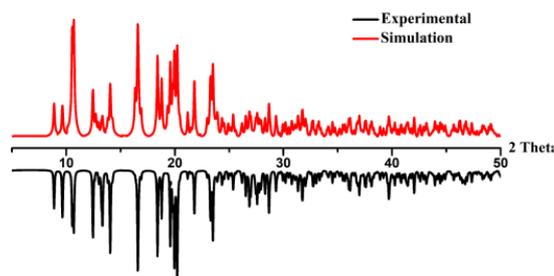


Fig. 5. PXRD patterns for the sample of 1

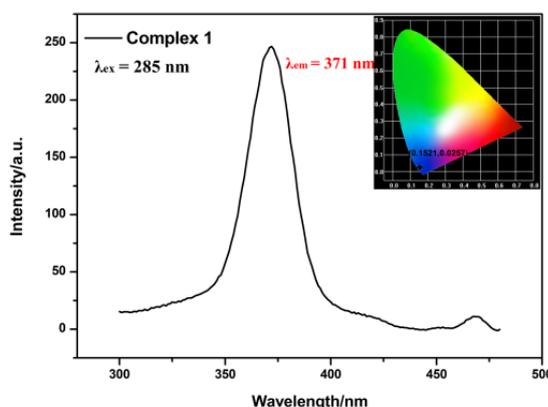


Fig. 6. Solid-state luminescence spectra of complex 1 at room temperature (inset: CIE chromaticity diagram)

4 CONCLUSION

We have employed 2-(1,2,4-triazol-1-yl)benzene-1,3,5-tricarboxylic acid (H_3L) as organic link for the divalent Cd(II) cation to construct complex $[Cd_5(\mu_5-HL^2-)_2(\mu_4-L^3-)_2(H_2O)_{10}] \cdot 2H_2O$ (**1**). Owing to the specific N/O coordination sites, the structural results are partially predictable. As might

have been expected, typical carboxylate groups bridge two Cd(II) ions to form a binuclear SBU unit. Furthermore, these units are linked by benzene carboxylate moieties to expand a 2D layer by ignoring the participation of N sites. From the view of 3D space, N-coordination promotes the development of the 3D framework. Besides, complex **1** has outstanding photoluminescence, specifically exhibiting blue fluorescence.

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