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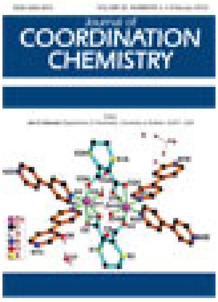

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A (3,9)-connected cadmium 1,2,4-triazolate framework based on a trinuclear inorganic cluster

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Hydrothermal reaction of 1,2,4-triazole with $CdCl_2 \cdot 2.5H_2O$ generated a 3-D metal-organic framework, $\{[Cd_3Cl_2(Trz)_4] \cdot H_2O\}_n$ (**1**) (Trz = 1,2,4-triazole), which was characterized by elemental analysis, FT-IR, X-ray powder diffraction, X-ray single-crystal diffraction, TG/DTA, and photoluminescence measurements. Compound **1** crystallizes in the orthorhombic system, space group *Pnma*, $a = 16.906(3)$ Å, $b = 8.3151(17)$ Å, $c = 13.080(3)$ Å, $V = 1838.6(6)$ Å^3, $Z = 4$. Cd(1) is coordinated by four nitrogen atoms and one chloride to form a distorted trigonal-bipyramidal geometry. Cd(2) is an octahedron defined by four triazole nitrogen atoms and two chlorides. Two Cd(2) and one Cd(1) are linked by μ_3-Cl(1) to give a [Cd_3Cl] cluster, which is connected by μ_2-Cl(2) to generate a 1-D inorganic chain. The 1-D inorganic chains are extended by μ_3-Trz to form a 2-D hybrid layer in the *b*, *c*-plane, which is ultimately linked by residual triazole ligands to give a 3-D framework. The [Cd_3Cl] clusters and the Trz ligands can be regarded as 9- and 3-connected nodes, which lead to an unusual (3,9)-connected net with Schläfli symbol of $(4^{23} \cdot 6^{13})(4^3)$. The solid **1** exhibits high thermal stability and shows strong blue fluorescence emission at 410 nm in the solid state at ambient temperature.

Keywords: Hydrothermal reaction; 1,2,4-Triazole; Crystal structure; Fluorescence; Topology

1. Introduction

In the past decade, metal-organic frameworks (MOFs) have been of interest owing to various architectures and exploitable applications as functional materials [1–5]. The diversity in framework structures of MOFs greatly depends on the selection of the metal centers and ligands, as well as on the reaction pathways. Many MOFs have been constructed from metal centers with well-defined coordination geometries and multifunctional organic ligands containing N- and/or O-donors [6–10]. Among the numerous ligands employed, 1,2,4-triazole and derivatives show particularly versatile coordination modes to bridge metal ions with potential $\mu_{1,2}$, $\mu_{2,4}$ and $\mu_{1,2,4}$ bridging modes that unite the coordination of both pyrazole and imidazole and exhibit an ability to afford polynuclear clusters and 1-D to 3-D MOFs [11–15]. For example, Su *et al.* [16] synthesized an exceptionably stable coordination polymer displaying open-ended,

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hollow, and nanotubular channels. Chen *et al.* [17–20] performed systematic work in this field and reported the solvothermal synthesis of a series of Cu(I) 1,2,4-triazolates involving *in situ* generation of triazole by cycloaddition of nitrile and ammonia.

The topological approach has proven to be an important and essential aspect in structural simplification and subsequent systematization of MOFs. The core idea of the work is to reduce multidimensional frameworks to simple node-and-linker reference nets [21–23]. A variety of uninodal networks have been reported [24–27]. However, higher-dimensional nets with mixed connectivity such as (3,6)-, (3,7)-, (4,8)-, (3,9)-, (3,10)-, and (3,12)-connected frameworks are difficult to achieve because of greater geometric limitations [28–32]. As a result, further investigation on the self-assembly of MOFs with mixed nodes will not only help facilitate the construction of MOFs with fascinating topologies but also enrich the growing database of coordination polymers.

As part of our ongoing research that deals with coordination chemistry of 1,2,4-triazoles, we have been focusing on discovering new high-connected MOFs with metal-1,2,4 triazolate polynuclear clusters as connectors. Using 1,2,4-triazole and anionic bridging ligands as coligands, a series of 6- to 10-connected 3-D MOFs based on binuclear, linear or cyclic trinuclear, tetranuclear, linear pentanuclear, cyclic hexanuclear, heptanuclear, and sixteen-nuclear metal–1,2,4-triazolate clusters have been obtained [33–36]. Detailed structural comparisons of the complexes indicate that the nuclearity of the metal–triazolate clusters and the bulk of the second bridging ligands are two key factors that influence the connectivity of the resultant 3-D MOFs. We now report a (3,9)-connected MOF based on $[Cd_3Cl]$ trinuclear building block, $\{[Cd_3Cl_2(Trz)_4]\cdot H_2O\}_n$ (**1**) (Trz = 1,2,4-triazole), which has been characterized by X-ray single-crystal diffraction, FT-IR spectrum, elemental analysis, X-ray powder diffraction (XRPD), TGA, and photoluminescence measurements.

2. Experimental

2.1. *Materials and physical measurements*

The reagents and solvents were commercially available and used without purification. The Fourier-transform infrared spectra (KBr pellets) were recorded using a Nicolet Avatar 360 FT-IR Spectrometer from 4000 to 400 cm^{-1}. C, H, and N elemental analyses were performed using an Elementar Vario EL III elemental analyzer. Thermal stability studies were carried out using a NETSCHZ STA−449C thermoanalyzer under a nitrogen atmosphere (40–1000°C range) at a heating rate of 5°C min^{-1}. The fluorescence spectra were measured with a Cary Eclipse fluorescence spectrophotometer at room temperature using powdered crystal samples. XRPD data were collected on a Bruker D8 Advance powder diffractometer with Cu-Kα radiation (40 kV, 40 mA). The simulated powder pattern was calculated using single-crystal X-ray diffraction data and processed by the free Mercury 2.3 program provided by the Cambridge Crystallographic Data Centre.

2.2. *Synthesis of* $\{[Cd_3Cl_2(Trz)_4]\cdot H_2O\}_n$

A mixture containing $CdCl_2\cdot 2.5H_2O$ (0.228 g, 1.0 mmol), 1,2,4-triazole (0.069 g, 1.0 mmol), and NaOH (0.04 g, 1.0 mmol) in 10 mL H_2O was heated at 180°C for 5 days under autogenous pressure, followed by cooling slowly (5°C h^{-1}) to room temperature. Pure colorless block crystals were collected by filtration, washed with water and ethanol, and dried in air. Yield: 0.13 g (*ca* 56% based on Cd). Anal. Calcd for $C_8H_{10}N_{12}OCl_2Cd_3$ (%): C, 13.76; H, 1.44; N, 24.07. Found (%): C, 13.58; H, 1.49; N, 25.03. FT-IR (solid KBr pellet/cm^{-1}): 3457(w), 2973(w), 2926(w), 1606(w), 1568(s), 1494(w), 1445(m), 1381(w), 1296(m), 1206(m), 1131(w), 1035(w), 985(w), 873(w), 752(s), 618(w).

2.3. *Crystal structure determination*

A single-crystal of **1** with dimensions $0.53 \times 0.42 \times 0.28\,mm^3$ was carefully selected under an optical microscope and glued to thin glass fibers. Crystallographic data for the compound were collected with a Siemens Smart CCD diffractometer with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71079$ Å) at $T = 293(2)$ K. Absorption corrections were made using *SADABS* [37]. The structure was solved using the direct method and refined by full-matrix least-squares on F^2 using the *SHELXL*-97 program package [38]. All non-hydrogen atoms were refined anistropically. Positions of the hydrogen atoms attached to carbons were fixed at their ideal positions. Crystal data as well as details of data collection and refinement for **1** are summarized in table 1. Selected bond lengths and angles are listed in table 2.

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

Empirical formula	$C_8H_{10}N_{12}OCd_3Cl_2$
Formula weight	698.38
Temperature (K)	293(2)
Crystal system	Orthorhombic
Space group	*Pnma*
Unit cell dimensions (Å, °)	
a	16.906(3)
b	8.3151(17)
c	13.080(3)
α	90
β	90
γ	90
Volume (Å^3), *Z*	1838.6(6), 4
Calculated density (g cm^{-3})	2.523
Absorption coefficient (mm^{-1})	3.755
F(000)	1312
θ range for data collection (°)	3.11–25.34
Reflections collected/unique	16,977/1807
R(int)	0.0276
Parameters	125
Goodness-of-fit on F^2	1.024
R_1[a], wR_2 [$I > 2\sigma(I)$]	0.0235, 0.0609
R_1, wR_2 (all data)	0.0242, 0.0613
Largest difference peak and hole (e Å^{-3})	0.907 and −0.521

[a] $R_1 = \sum(|F_o| - |F_c|)/\sum|F_o|$; $wR_2 = [\sum w(F_o^2 - F_c^2)^2/\sum w(F_o^2)^2]^{0.5}$.

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

Cd(1)–N(2)	2.226(4)	Cd(2)–N(3)	2.309(3)
Cd(1)–N(5)	2.253(3)	Cd(2)–N(7)	2.340(3)
Cd(1)–N(6)	2.348(4)	Cd(2)–N(4)	2.353(3)
Cd(1)–Cl(1)	2.6631(13)	Cd(2)–Cl(1)	2.7228(9)
Cd(2)–N(1)	2.302(3)	Cd(2)–Cl(2)	2.6472(9)
N(2)–Cd(1)–N(5)	133.23(9)	N(1)–Cd(2)–Cl(2)	92.94(9)
N(5)#1–Cd(1)–N(5)	93.49(18)	N(3)–Cd(2)–Cl(2)	91.60(8)
N(2)–Cd(1)–N(6)	85.29(17)	N(7)–Cd(2)–Cl(2)	88.29(8)
N(5)–Cd(1)–N(6)	92.02(11)	N(4)–Cd(2)–Cl(2)	93.00(8)
N(2)–Cd(1)–Cl(1)	87.73(12)	N(1)–Cd(2)–Cl(1)	87.67(8)
N(5)–Cd(1)–Cl(1)	92.76(8)	N(3)–Cd(2)–Cl(1)	87.80(8)
N(6)–Cd(1)–Cl(1)	173.02(12)	N(7)–Cd(2)–Cl(1)	92.44(8)
N(1)–Cd(2)–N(3)	175.40(11)	N(4)–Cd(2)–Cl(1)	86.28(8)
N(1)–Cd(2)–N(7)	90.60(11)	Cl(2)–Cd(2)–Cl(1)	179.05(3)
N(3)–Cd(2)–N(7)	88.77(11)	Cd(1)–Cl(1)–Cd(2)	101.48(3)
N(1)–Cd(2)–N(4)	88.52(11)	Cd(2)#1–Cl(1)–Cd(2)	100.73(4)
N(3)–Cd(2)–N(4)	92.00(11)	Cd(2)#2–Cl(2)–Cd(2)	102.24(5)
N(7)–Cd(2)–N(4)	178.48(11)		

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

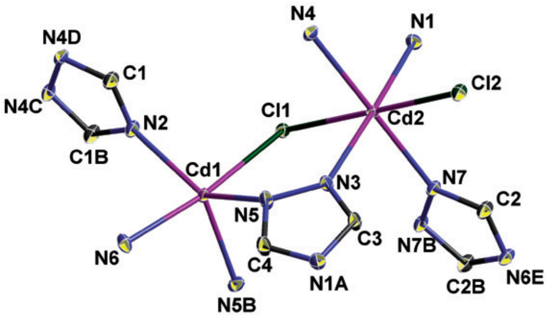

Figure 1. View of the coordination environments in **1** (symmetry codes: (A) $0.5-x$, $-y$, $-0.5+z$; (B) x, $0.5-y$, z; (C) $-x$, $0.5+y$, $2-z$; (D) $-x$, $-y$, $2-z$; (E) $0.5+x$, $0.5-y$, $1.5-z$).

3. Results and discussion

3.1. *Description of crystal structure*

Single-crystal structure analysis shows that **1** crystallizes in the orthorhombic space group *Pnma* and can be described as an unprecedented (3,9)-connected framework based on [Cd$_3$Cl] trinuclear clusters and μ_3-Trz ligands. The asymmetric unit of **1** consists of two crystallographically unique Cd^{2+} ions, three independent trz ligands (Trz-I, C(1)N(2)C(1B)N(4C)N(4D); Trz-II, C(3)N(1A)C(4)N(5)N(3); Trz-III, C(2)N(6E)C(2B)N(7B)N(7)), two chlorides, and one crystalline H$_2$O (figure 1). The independent Cd(1) is five-coordinate to four nitrogen atoms from triazoles and one chloride to construct a distorted trigonal-bipyramidal geometry. Cd(2) connects four triazole nitrogen atoms and two chlorides to give an octahedral [CdCl$_2$N$_4$] geometry. The Cd–N and Cd–Cl bond lengths are 2.226(4)–2.353(3) Å and

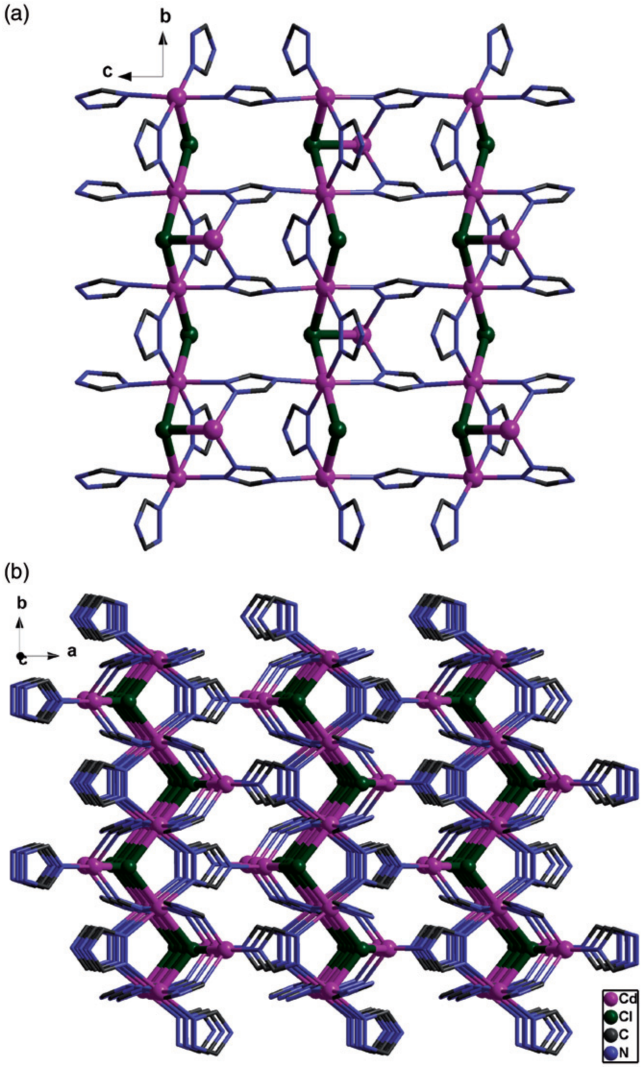

Figure 2. (a) The 2-D layer generated by 1-D Cd–Cl chain in the *b*, *c*-plane. (b) The 3-D framework of **1** viewed along the *c*-axis.

2.6472(9)–2.7228(9) Å, respectively. The corresponding bond angles are 85.29(17)–179.05(3)°. One Cd(1) and two Cd(2) are linked by μ_3-Cl(1) to form a [Cd_3Cl] trinuclear cluster, which is further connected by one μ_2-Cl(2) generating a 1-D [Cd_3Cl_2]$_n$ inorganic chain along the *b*-axis. Adjacent Cd–Cl inorganic chains are bridged by μ_3-Trz-II to form a 2-D layer in the *b*, *c*-plane (figure 2a), which is further extended by Trz-I and Trz-III to generate a 3-D metal-organic hybrid framework (figure 2b). In our analysis of the 3-D framework, we can define the [Cd_3Cl] trinuclear cluster as a node. Each [Cd_3Cl] cluster links three Trz-I ligands and six adjacent trinuclear motifs through four Trz-II and two Trz-III (figure 3a). Thus, the [Cd_3Cl] clusters and Trz-I ligands can be regarded as 9- and 3-conneted nodes, respectively (figure 3b). These two non-equivalent nodes

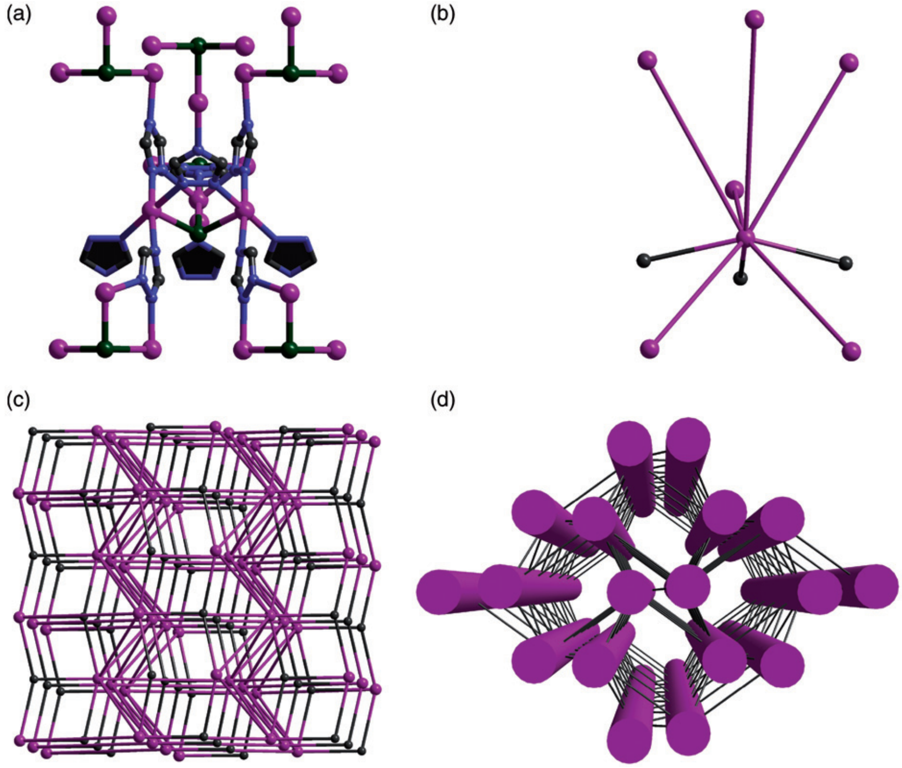

Figure 3. (a) The linkage of each [Cd_3Cl] trinuclear unit with three Trz-I (highlighted by the gray plane) and six adjacent trinuclear motifs. (b) The 9-connected node. (c) The schematic presentation of the (3,9)-connected topological net. (d) The rod-packing framework of **1**.

lead to an unusual (3,9)-connected network topology (figure 3c). The node-node distances are 4.31, 4.35, 8.38, and 10.39 Å. The short Schläfli symbol for the net is $(4^{23}\cdot 6^{13})(4^3)$, which represents the 9-connected [Cd_3Cl] node and 3-connected Trz-I node, respectively. The long Schläfli symbols are $4\cdot 4\cdot 4\cdot 4\cdot 4\cdot 4\cdot 4\cdot 4\cdot 4\cdot 4\cdot 4\cdot 4\cdot 4\cdot 4\cdot 4\cdot 4^2\cdot 4^2\cdot 4^2\cdot 4^2\cdot 4^2\cdot 4^3\cdot 4^3\cdot 6^2\cdot 6^3\cdot 6^3\cdot 6^3\cdot 6^7\cdot 6^7\cdot 6^8\cdot 6^8\cdot 6^8\cdot 6^8\cdot 6^8\cdot 6^8\cdot 6^8$ for trinuclear cluster and $4^2\cdot 4^3\cdot 4^3$ for Trz-I. Alternatively, Yaghi *et al.* [39] recently demonstrated a concept of infinite rod-like secondary building units in the design and synthesis of MOFs. According to the concept, the 3-D net of **1** exhibits a rather simple topological framework based on 1-D rod-shaped $[Cd_3Cl_2]_n$ building blocks. Each 1-D rod shaped building block links five adjacent rods through three independent triazole ligands to generate the 3-D rod-packing structure of **1** (figure 3d). The example demonstrates again the usefulness of the concept of rod packing in simplifying the metal-organic coordination frameworks.

3.2. *XRPD, TGA, and photoluminescence properties*

Compound **1** was characterized *via* XRPD at room temperature (figure 4a). The XRPD pattern measured for the sample was in good agreement with the XRPD pattern

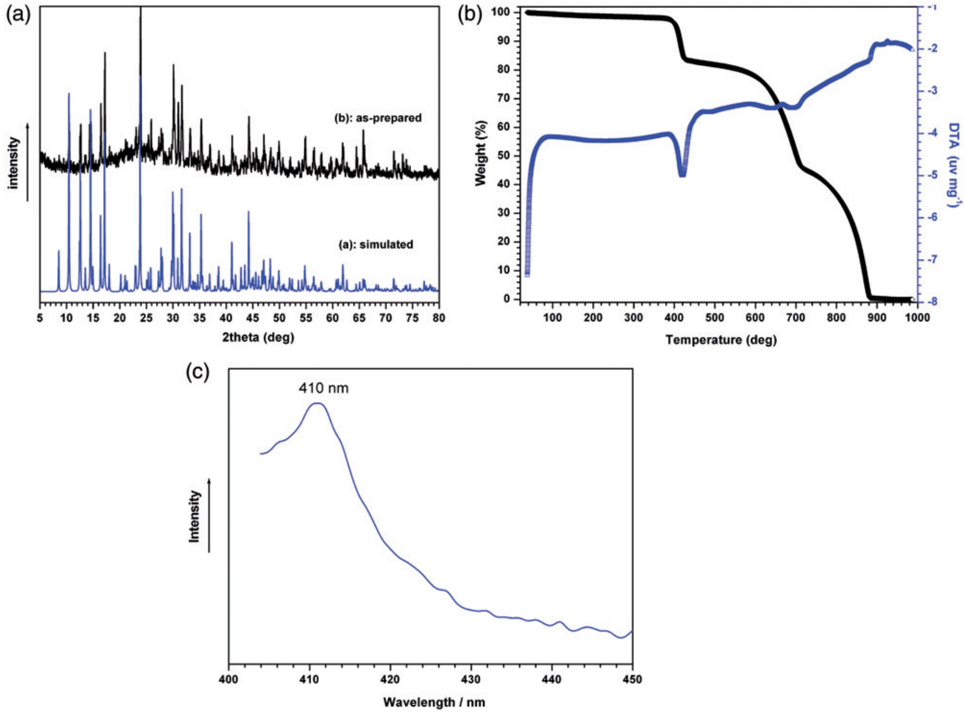

Figure 4. (a) XRPD patterns for **1**: top, calculated from single-crystal X-ray data; bottom, experimental data. (b) TG/DTA curves of **1**. (c) Solid-state emission spectrum of **1** at ambient temperature.

simulated from the single-crystal X-ray data using Mercury 1.4. On the basis of the XRPD results, it can be established that the single-crystal selected is a good representative of the bulk compound.

The thermal stability of **1** was investigated on crystalline samples under N_2 from 40°C to 1000°C using the TG/DTA analyses. The weight loss curve of **1** showed that it was stable to *ca* 380°C (figure 4b). The slight weight loss before 380°C is due to loss of one crystallization water (exptl: 2.2%, Calcd: 2.6%). In the range 380−710°C, two continuous mild weight losses are due to decomposition of four triazoles and two Cl (exptl: 50.9%, Calcd: 49.1%). In the temperature range 720–900°C, a sharp weight loss process is ascribed to the sublimation of formed Cd metal (exptl: 46.9%, Calcd: 48.3%).

We also studied the photoluminescence of **1** at room temperature. Upon excitation at 301 nm, an intense band in the emission spectrum was observed at 410 nm (figure 4c). To understand more thoroughly the nature of this emission band, the luminescence of Trz was also investigated. The ligand is nearly non-fluorescent from 400 to 800 nm for excitation wavelengths between 250 and 450 nm. However, the highest occupied molecular orbitals (HOMOs) in **1** are presumably associated with the π-bonding orbitals from the aromatic 1,2,4-triazole rings, whereas the lower unoccupied molecular orbitals (LUMOs) are associated mainly with Cd–Cl δ*-antibonding orbitals, being localized more on the metal centers. Thus, the origin of the weak emission can be attributed to ligand-to-metal charge transfer (LMCT) [40].

4. Conclusion

A (3,9)-connected Cd-triazolate framework constructed from trinuclear clusters has been synthesized and characterized. High-connected MOFs based on polynuclear metal–1,2,4-triazolate motifs continue to be investigated in our lab.

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