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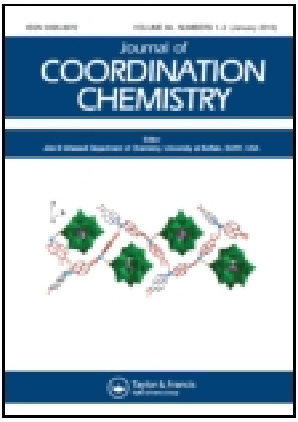

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Two Cd(II) complexes derived from mercapto-triazole ligands: syntheses, crystal structures, and luminescent properties

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Two Cd(II) complexes derived from mercapto-triazole ligands: syntheses, crystal structures, and luminescent properties

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Two cadmium complexes, $\{[Cd(a\text{-}ptt)(ptt)]\cdot H_2O\}_n$ (**1**) and $[Cd(a\text{-}Hmtt)_2(SO_4)H_2O]\cdot CH_3OH$ (**2**), have been prepared based on 4-amino-3-(4-pyridine)-5-mercapto-1,2,4-triazole (a-Hptt) and 4-amino-3-methyl-5-mercapto-1,2,4-triazole (a-Hmtt), respectively. In **1**, amino-triazole ligand a-Hptt can partly be deaminated and transformed into 3-(4-pyridine)-5-mercapto-triazole (Hptt) under hydrothermal conditions. X-ray diffraction analysis reveals that **1** exhibits an unusual 2-D lamp-shade-type layer structure in which the amino ligand a-ptt and the deamination ligand ptt display *exo*-tridentate bridging and bidentate bridging, respectively. Complex **2** is mononuclear and further assembled into a 3-D supramolecular architecture via non-covalent interactions. Complexes **1** and **2** were characterized by elemental analyses, IR, and thermogravimetric analyses. Furthermore, solid-state luminescent properties of **1** and **2** have also been investigated.

Keywords: Mercapto-triazole ligands; Deamination; Cd(II) complexes; Crystal structures; Luminescent properties

1. Introduction

Metal-organic frameworks (MOFs) display fascinating structures [1–5] and useful properties in photoelectrical [6, 7], magnetic [8], catalytic [9], and porous materials [10]. Although some results have been achieved [11–13], it is still a challenge to rationally and predictably assemble MOFs. In construction of MOFs, investigating the factors influencing assembly of MOFs is important because different conditions can affect ultimate structural motifs. The main factors affecting the final assembly are ligands, metal centers, reaction temperature, solvents, pH, etc. [14–18]. Selections of organic ligands with versatile binding modes and metal centers have proved to be key to construction of desirable structures.

Triazole and its derivatives which combine the coordination geometries of both pyrazole and imidazole have been studied as bridging ligands with several electron-rich donor centers. Many triazole-based mononuclear, oligonuclear, and polynuclear MOFs have been reported [19–22]. However, syntheses and crystallographic studies on MOFs based on

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*Corresponding authors. Email: lilinke@zzu.edu.cn (L. Li); houhongw@zzu.edu.cn (H. Hou) pyridine containing mercapto-triazole are limited [23, 24]. Thus, further efforts are required to extend knowledge of the construction of various coordination complexes containing mercapto-triazole bridging ligands with intriguing structures and useful properties.

The d^{10} metal ion often selected for the spherical configuration possesses variable coordination geometry and is suited for construction of multiform coordination complexes and networks [25, 26]. Interesting luminescence properties of d^{10} metal complexes such as Cd(II), Hg(II), and Zn(II) have been reported [27–29].

To evaluate the effect of different substituent groups in triazole rings (e.g. methyl and pyridine) on the design and assembly of MOFs, we have selected 4-amino-3-(4-pyridine)-5mercapto-1,2,4-triazole (a-Hptt) and 4-amino-3-methyl-5-mercapto-1,2,4-triazole (a-Hmtt) (scheme 1) as ligands to assemble with Cd(II). In **1**, amino-triazole ligand a-Hptt can be deaminated and transformed into 3-(4-pyridine)-5-mercapto-triazole (Hptt, scheme 1) under hydrothermal conditions. Herein, we report syntheses and characterizations of a 2-D lampshade-type layer structure Cd(II) complex $\{[Cd(a\text{-}ptt)(ptt)]\cdot H_2O\}_n$ (**1**) and a mononuclear Cd(II) complex $[Cd(a\text{-}Hmtt)_2(SO_4)(H_2O)]\cdot CH_3OH$ (**2**). Additionally, **1** and **2** show thermal stabilities and **1** displays intense luminescent emission at room temperature, while **2** exhibits fluorescence quenching.

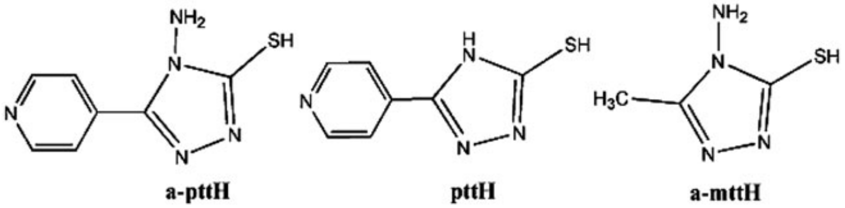
2. Experimental

2.1. *General methods*

The a-Hptt and a-Hmtt were prepared according to reported procedures [30, 31]. All other chemicals were from commercial sources without purification. Elemental analyses (C, H, and N) were carried out with a Carlo-Erba1106 elemental analyzer. IR data were recorded with a BRUKER TENSOR 27 spectrophotometer with KBr pellets from 400 to 4000 cm^{-1}. TG-DSC measurements were performed by heating the crystalline sample from 20 to 800 °C at 10 $K\,min^{-1}$ in air with a Netzsch STA 409PC differential thermal analyzer. The fluorescence properties of the complexes were measured with a F-4500 FL spectrophotometer.

2.2. *Syntheses*

2.2.1. Synthesis of 1. A mixture of $Cd(NO_3)_2\cdot 4H_2O$ (9.3 mg, 0.03 mM), a-Hptt (11.6 mg, 0.06 mM), DMF (3 mL), and distilled water (5 mL) was sealed in a Teflon-lined stainless-steel autoclave. The mixture was heated at 100 °C for three days and then cooled



Scheme 1. Chemical structure of the ligands a-pttH, pttH and a-mttH.

to room temperature at 5 °C/h; pale yellow block-shaped crystals of **1** were obtained. Yield: 62%. Anal. Calcd (%) for **1**: C, 33.84; H, 2.03; N, 25.37. Found: C, 33.42; H, 2.33; N, 26.03. IR (KBr, cm^{-1}): 1610.89(s), 1541.74(w), 1467.12(w), 1415.45(s), 1377.69 (s), 1222.42(m), 1160.82(m), 1087.49(w), 1061.53(w), 1009.87(m), 962.41(w), 831.83(m), 714.73(m), 698.81(m), 688.69(m), 602.70(w), 519.90(w).

2.2.2. Synthesis of 2. DMF solution (1 mL) of a-Hmtt (7.9 mg, 0.06 mM) was added dropwise to an aqueous solution (3 mL) of $CdSO_4 \cdot 8/3H_2O$ (15.4 mg, 0.06 mM) and then methanol solution (3 mL) was slowly added into the mixture. The resulting mixture was left to stand undisturbed at room temperature. After one week, colorless block crystals suitable for X-ray analysis were obtained. Yield: 78%. Anal. Calcd (%) for **2**: C, 16.46; H, 1.97; N, 21.94. Found: C, 16.25; H, 1.89; N, 22.03. IR (cm^{-1}, KBr): 3386.45(m), 3122.84 (s), 2957.99(m), 2777.38(w), 1660.26(m), 1627.60(w), 1587.63(w), 1499.65(s), 1396.35 (w), 1352.86(m), 1213.33(m), 1139.41(s), 1090.21(s), 1058.05(s), 989.34(m), 748.29(w), 671.21(w), 618.76(m), 605.41(m), 471.37(w).

2.3. *X-ray crystallographic determinations for 1 and 2*

Crystal data collection and refinement parameters for **1** and **2** are given in table 1. All measurements were made with a Bruker Apex CCD diffractometer with graphite monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Single crystals were selected and mounted on a glass fiber. The data were collected at 291(2) K and corrected for Lorentz-polarization effects. A correction for secondary extinction was applied. The structures were solved by direct methods and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogens were included but not refined. The final cycle of

Table 1. Crystallographic data for **1** and **2**.

Complexes	**1**	**2**
Formula	$C_{14}H_{10}CdN_9OS_2$	$C_{3.50}H_5Cd_{0.50}N_4O_3S_{1.50}$
fw	496.83	255.40
Crystal system	Monoclinic	Orthorhombic
Space group	*P2₁/c*	*Pnma*
a (Å)	10.737(2)	8.1553(16)
b (Å)	18.368(4)	14.987(3)
c (Å)	9.4217(19)	13.615(3)
α (°)	90	90
β (°)	101.15(3)	90
γ (°)	90	90
V (Å^3)	1823.0(6)	1664.0(6)
Z	4	8
D_c (g cm^{-3})	1.810	2.039
F (000)	980.0	1008.0
θ range for data collection (deg)	2.47–25.00	2.91–27.50
Reflections collected/unique	17,279/3198	17,266/1974
Data/restraints/parameters	3198/0/245	1974/0/127
Goodness-of-fit on F^2	1.080	1.075
R_1[a] = [$I > 2\sigma(I)$]	0.0463	0.0296
wR_2[b] (all data)	0.1155	0.0766

[a]: $R_1 = ||F_o| - |F_c||/|F_o|$. [b]: $wR_2 = [w(|F_o^2| - |F_c^2|)^2/w|F_o^2|^2]^{1/2}$. For **1** $w = 1/[\sigma^2(F_o)^2 + 0.0473P^2 + 5.8327P]$, where $P = (F_o^2 + 2F_c^2)/3$. For **2** $w = 1/[\sigma^2(F_o)^2 + 0.0333P^2 + 4.6688P]$, where $P = (F_o^2 + 2F_c^2)/3$.

full-matrix least-squares refinement was based on observed reflections and variable parameters. All calculations were performed using the SHELXL-97 crystallographic software package. Selected bond lengths and angles are tabulated in table 2.

3. Results and discussion

3.1. *Descriptions of crystal structures*

Single-crystal X-ray diffraction analysis reveals that **1** crystallizes in the monoclinic space group $P2_1/c$ and exhibits an attractive 2-D lampshade-type layer structure. As illustrated in figure 1(a), Cd(II) is five-coordinate in a slightly distorted square-pyramidal geometry, surrounded by S2 donor on the apical site, two N_{trz} (trz = triazole), one N_{py}, and the S1 donors in the basal plane. In **1**, the amino a-ptt provides one N_{trz} (trz = triazole), one N_{py}, and one sulfur to coordinate with the central Cd(II) in an *exo*-tridentate bridge, while the deamination ligand ptt only offers one N_{trz} and one sulfur in a bidentate bridge. The Cd–N bond lengths are 2.319(4)–2.364(5) Å (table 1), close to those observed previously in related Cd(II) complexes [32, 33]. The Cd–S bond distances, 2.677(2) and 2.585(2) Å, are comparable to the distance of 2.623(2) Å found in $[Cd(L5)_2]_n$ (L5 = 5-(2-pyridyl)-1,3,4-oxadiazole-2-thione) [34]. The bond angles around Cd(II) range from 80.04(12) to 162.98 (11)°.

Neighboring complex units are connected to assemble a 2-D lampshade-type layer structure. When we neglect bidentate ptt ligands and regard the *exo*-tridentate a-ptt groups as "nodes," a 2-D (6,3) coordination layer extended along the *ac* plane can be distinctly observed (figure 1(b)). The Cd···Cd distances are 10.737(2), 4.738(2), and 10.871(3) Å in

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

Complex **1**			
Cd(1)–N(6)#1	2.319(4)	N(10)#2–Cd(1)–N(1)#3	96.97(15)
Cd(1)–N(10)#2	2.331(4)	N(10)#2–Cd(1)–S(2)	101.71(12)
Cd(1)–N(1)#3	2.364(5)	N(6)#1–Cd(1)–S(1)	92.19(11)
Cd(1)–S(1)	2.6769(16)	N(1)#3–Cd(1)–S(1)	162.98(11)
Cd(1)–S(2)	2.5848(15)	N(6)#1–Cd(1)–N(10)#2	149.50(16)
N(6)#1–Cd(1)–N(1)#3	81.91(15)	N(1)#3–Cd(1)–S(2)	96.98(11)
N(6)#1–Cd(1)–S(2)	108.70(12)	N(10)#2–Cd(1)–S(1)	80.04(12)
N(6)#1–Cd(1)–N(10)#2	149.50(16)	S(2)–Cd(1)–S(1)	100.04(5)
Complex **2**			
Cd(1)–O(1)	2.338(3)	Cd(1)–O(4)	2.379(3)
Cd(1)–N(4)	2.477(2)	Cd(1)–N(4)#1	2.477(2)
Cd(1)–S(1)	2.5744(9)	Cd(1)–S(1)#1	2.5744(9)
O(1)–Cd(1)–O(4)	171.43(11)	O(1)–Cd(1)–N(4)	93.08(8)
O(4)–Cd(1)–N(4)	80.27(9)	O(1)–Cd(1)–N(4)#1	93.08(8)
O(4)–Cd(1)–N(4)#1	80.27(9)	N(4)–Cd(1)–N(4)#1	77.64(10)
O(1)–Cd(1)–S(1)	95.40(4)	O(4)–Cd(1)–S(1)	88.67(5)
N(4)–Cd(1)–S(1)	78.94(5)	N(4)#1–Cd(1)–S(1)	155.47(5)
O(1)–Cd(1)–S(1)#1	95.40(4)	O(4)–Cd(1)–S(1)#1	88.67(5)
N(4)–Cd(1)–S(1)#1	155.47(6)	N(4)#1–Cd(1)–S(1)#1	78.94(5)
S(1)–Cd(1)–S(1)#1	122.93(4)	N(3)–N(4)–Cd(1)	110.30(15)
S(2)–O(1)–Cd(1)	113.08(15)	C(1)–S(1)–Cd(1)	97.68(10)

Note: Symmetry transformations used to generate equivalent atoms: For **1**: #1 x, $-y+1/2$, $z+1/2$; #2 $x+1$, $-y+1/2$, $z+1/2$; #3 x, $-y+1/2$, $z-1/2$; #4 $x-1$, $-y+1/2$, $z-1/2$. For **2**: #1 x, $-y+3/2$, z.

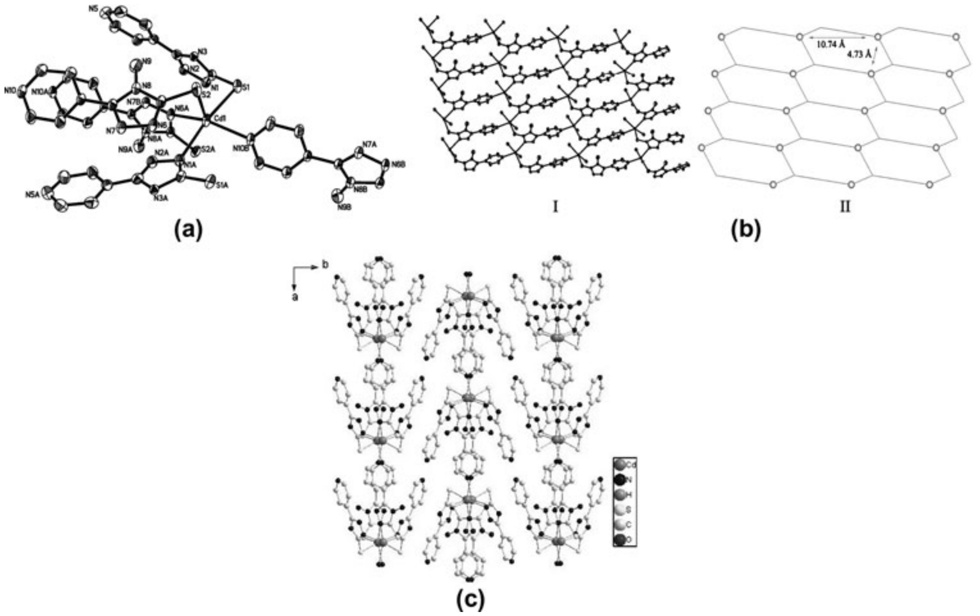


Figure 1. (a) Coordination environment of Cd(II) in **1**. Ellipsoids are drawn at the 30% probability level with all hydrogens omitted for clarity. (b) I: The 2-D (6,3)-connected structure of **1** along the *ac* plane with all bidentate a-ptt omitted for clarity. II: Schematic illustration of a single net in **1** (circles: Cd(II)). (c) Lampshade-type structure of **1**.

the *a*-, *c*-, and *ac*- directions, respectively. The most striking feature of **1** is the lampshade-type configuration of each layer along the *a*-axis. Bidentate bridging ptt ligands containing uncoordinated N_{py} are decorated on left and right of the layers. Adjacent lampshade-type structures alternately arrange in the ···ABAB···sequence and the closest Cd···Cd distance in the adjacent layers is 10.380(2) Å (figure 1(c)). Adjacent 2-D layers are further extended to a 3-D framework via two types of weak hydrogen bonding interactions between H_2O molecules and the pyridine C (C–H···O 3.35 Å), as well as N_{py} atom (O···N_{py} 2.873(9) Å); the existence of these hydrogen bonding interactions stabilizes the solid-state structure of **1**.

X-ray analysis reveals that **2** crystallizes in the orthorhombic space group *Pnma*. In contrast to **1**, **2** is mononuclear (as depicted in figure 2(a)), the center Cd(II) is six-coordinate in a stretched octahedral geometry with two nitrogens from two triazoles, two sulfurs from two a-Hmtt, and two oxygens from sulfate and water, respectively. In **2**, two nitrogens and two sulfurs together with the central Cd(II) form the equatorial plane (mean deviation from the plane is 0.0082 Å) and the remaining two oxygens are axial. In the equatorial plane, the Cd–N bond length (2.477(2) Å) is somewhat greater than those (2.319(4)–2.364(5) Å) of **1**, while the Cd–S bond length (2.574(9) Å) is slightly shorter than those (2.585(2)–2.677(2) Å) of **1**. N4A–Cd1–S1 and S1–Cd1–S1A bond angles are 155.47(6)° and 122.93(4)°, the N4–Cd1–S1 and N4–Cd1–N4A bond angles are 78.94(5)° and 77.64(10)°, respectively. In the axial positions, the Cd1–O1 and Cd1–O4 bond lengths are 2.338(3) and 2.379(3) Å, respectively, and the O1–Cd1–O4 bond angle is 171.43(11)°.

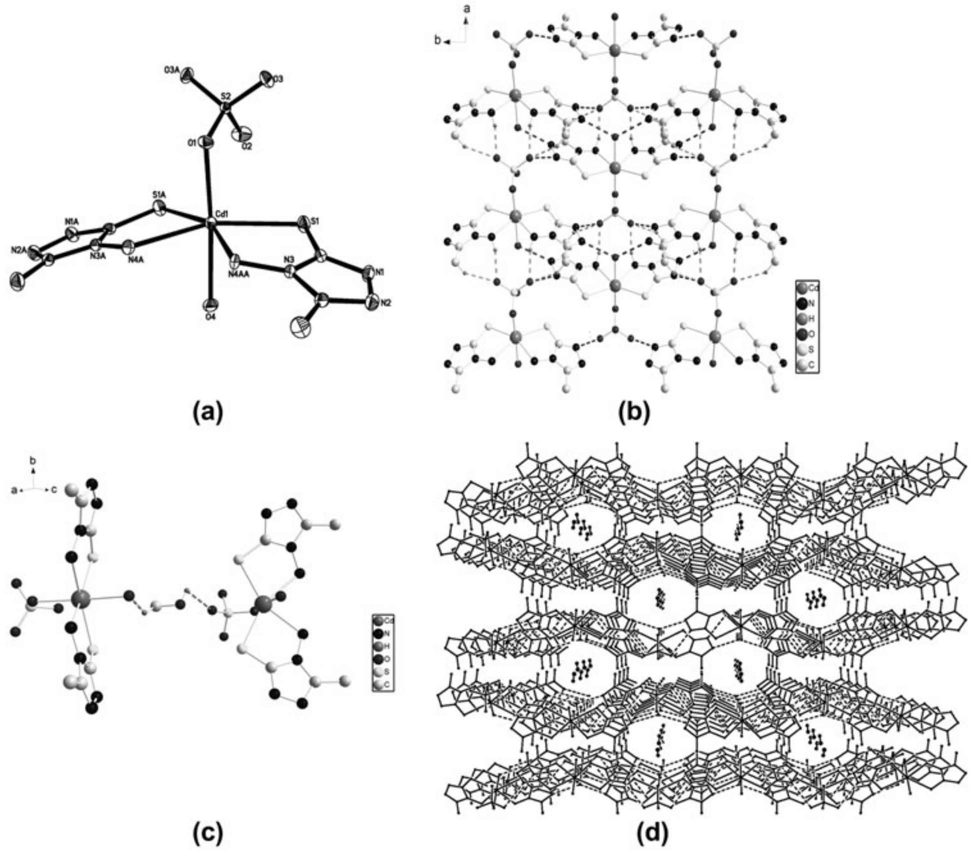


Figure 2. (a) Coordination environment around Cd(II) of **2**. (b) View of the 2-D supramolecular structure of **2** by non-covalent interactions along the *c*-axis. (c) Hydrogen bonds (C–H···O and O–H···O) between the host compound and guest methanol in **2**. (d) The solid-state structure of **2**.

In the crystal structure, mononuclear units are connected by N–H···O interactions (amine/sulfate) with N···O distance of 2.997(4) Å, O···N (water/triazole) with distance of 2.869(3) Å, N···O (triazole/sulfate) with distance of 2.890(4) Å, and C–H···O (methyl/sulfate) with C···O distance of 3.341(4) Å, giving a 2-D layer (figure 2(b)). The N···O and C···O distances are comparable to those observed in other complexes [35, 36]. In addition, free methanols are found in the lattice of **2** with intermolecular hydrogen bonding interactions between free methanol molecules, coordinated water and sulfate with the C···O (methanol/water) distance of 3.414(12) Å and the O···O (methanol/sulfate) distance of 2.758(9) Å (figure 2(c)), respectively. The C···O and O···O distances are both within the ranges of those observed in other complexes [37, 38]. The 2-D layers are further extended via these hydrogen bonding interactions into a 3-D metal-organic supramolecular network (figure 2(d)).

Comparing the conformation of **1** with **2**, the substituent groups are important in the final assemblies of the complexes. In **1**, due to the presence of pyridine, a-Hptt displays two different bridges and connects adjacent Cd(II) ions to form a 2-D lampshade-type layer structure. However, in **2**, methyl containing mercapto-triazole a-Hmtt is only bidentate chelating SO_4^{2-} coordinates generating a mononuclear structure.

3.2. *Thermal analyses*

Thermal stability of **1** and **2** were determined using a TG-DSC analyzer. **1** shows weight loss of 3.64% (Calcd 3.63%) from 70 to 165 °C, assigned to the loss of one water (Supplementary material). Decomposition of organic components of **1** occurs from 240 to 770 °C, indicating destruction of the 2-D network. After that, no further mass loss was observed, and the final product may be ascribed to CdS (obsd. 28.16%, Calcd 29.08%). In the DSC curve, three endothermic peaks appear at 297, 318, and 500 °C. Complex **2** is stable to 80 °C. From 80 to 170 °C, it lost 6.12%, assigned to loss of one methanol (6.03%). Then, there is a distinct weight loss of 3.52% from 213 to 220 °C, corresponding to release of one coordinated water (3.44%). The residual sample starts to gradually decompose with weight loss ending at 710 °C. Upon further heating to 900 °C, no weight loss is observed and the final product may be CdO (obsd. 27.08%, Calcd 26.96%). Correspondingly, three endothermic peaks are in the DSC curve at 271, 378, and 704 °C, respectively.

3.3. *Photoluminescence properties*

Coordination complexes of metal cations with d^{10} configuration have interesting luminescent properties [27–29, 39]. In this work, the luminescence of **1**, **2,** and the corresponding free ligands were investigated in the solid state at room temperature. For **1**, as indicated in figure 3, the free a-Hptt exhibits broad weak luminescent emission with three emission peaks at 468, 486, and 542 nm ($\lambda_{ex} = 300$ nm), respectively, which can be ascribed to the π–π^* and/or n–π^* transitions. In contrast, **1** shows broad strong emission with peaks occurring at 460, 483, and 531 nm ($\lambda_{ex} = 300$ nm), respectively. The similarity of the emission bands of **1** with that of free a-Hptt allows assignment to the intraligand fluorescent emission [40]. It is, therefore, conceivable that the slight blue-shift of **1** relative to free a-Hptt results from the effect of metal-ligand coordination interactions. Moreover, the luminescent intensity of **1** is almost fivefold larger than that of a-Hptt, from coordination of the ligands to Cd(II) increasing the rigidity of the molecular edifice, resulting in a decrease in the non-radiative decay of the intraligand π–π^* excited state [41].

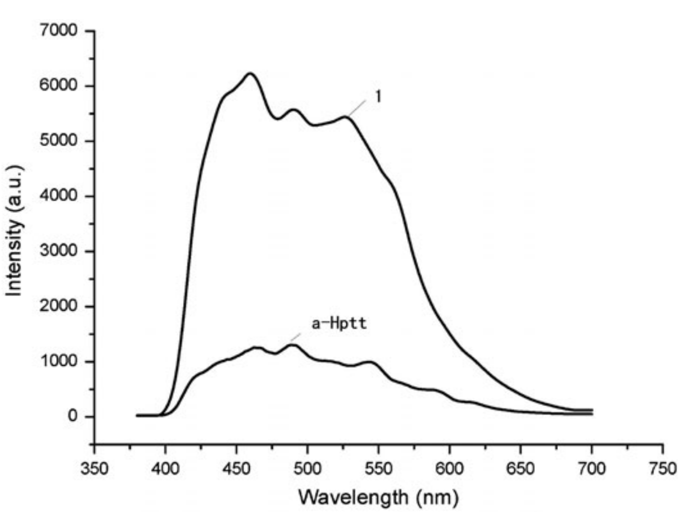


Figure 3. The solid-state fluorescence spectra of **1** and a-Hptt at room temperature.

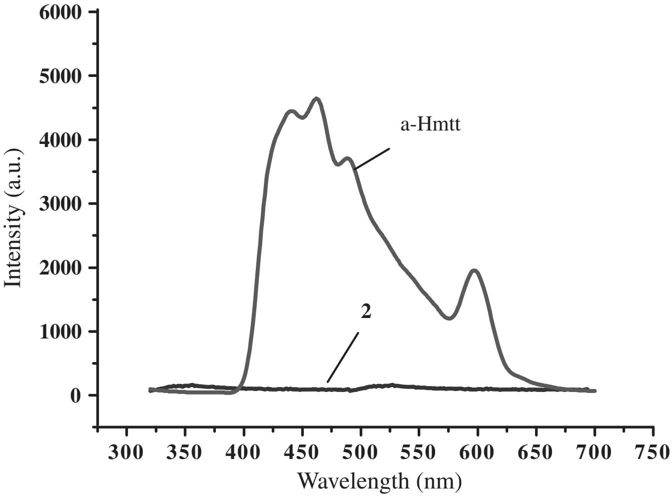


Figure 4. The solid-state fluorescence spectra of **2** and a-Hmtt at room temperature.

For **2**, as shown in figure 4, free a-Hmtt shows an emission maximum at 464 nm ($\lambda_{ex} = 300$ nm). In comparison with free ligand, **2** exhibits fluorescence quenching, assigned to coordination of solvent [42, 43], which can lead to the energy transfer not carried out perfectly.

4. Conclusions

This study investigated the syntheses and structures of metal organic framework using Cd(II) and mercapto-triazole ligands (a-Hptt and a-Hmtt) under hydrothermal condition and room temperature, respectively. An unusual 2-D lampshade-type layer coordination polymer **1** and a mononuclear **2** have been obtained. **1** shows deamination-assisted structural modification due to a part of a-ptt being deaminated and transformed into ptt under hydrothermal conditions. **2** exhibits non-covalent interactions. Complex **1** exhibits strong fluorescence in the solid state at room temperature so that it could be potentially used as a luminescent material.

Supplementary material

Crystallographic data for the structural analysis has been deposited with the Cambridge Crystallographic Data Center. CCDC 881607 and 914510 contain supplementary crystallographic data for **1** and **2**. The data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.htm (or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336033).

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