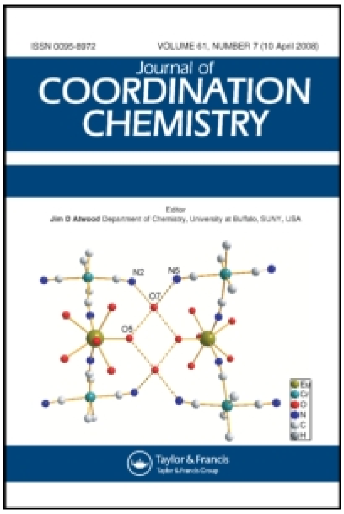

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

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To cite this Article Li, Wei , Li, Ming-Xing , Yang, Jia-Jun , Shao, Min and Liu, Hong-Jiang(2008) 'Cu(II)/Cd(II) coordination polymers based on polycarboxylate and 2-(2-pyridyl)benzimidazole', Journal of Coordination Chemistry, 61: 17, 2715 — 2724

To link to this Article: DOI: 10.1080/00958970801958980

URL: http://dx.doi.org/10.1080/00958970801958980

Journal of Coordination Chemistry
Vol. 61, No. 17, 10 September 2008, 2715–2724

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Cu(II)/Cd(II) coordination polymers based on polycarboxylate and 2-(2-pyridyl)benzimidazole

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(*Received 20 June 2007; in final form 27 September 2007*)

Three polycarboxylate coordination polymers containing 2-(2-pyridyl)benzimidazole as co-ligand, $[Cu(Bdc)(2\text{-}PyBIm)]_n$ (**1**), $[Cu(HBtc)(2\text{-}PyBIm)]_n$ (**2**) and $[Cd_2(HBtc)_2(2\text{-}PyBIm)_2]_n \cdot nH_2O$ (**3**) ($H_2Bdc = 1,4$-benzenedicarboxylic acid; $H_3Btc = 1,3,5$-benzenetricarboxylic acid), have been synthesized and characterized by elemental analyses, IR spectra, TG-DSC and X-ray structural analyses. Complex **1** is a one-dimensional zigzag chain in which the Cu(II) is *cis* six-coordinated by two chelating carboxyl groups and a 2-PyBIm ligand. Complex **2** is a two-dimensional (4, 4) network in which H_3Btc is partially deprotonated. Complex **3** has a three-dimensional framework in which one Cd(II) is six-coordinate and the other is seven-coordinate. All 2-PyBIm groups are neutral, chelating, bidentate ligands in **1**–**3**. These complexes are quite thermally stable. The luminescence of **3** has also been investigated.

Keywords: 2-(2-Pyridyl)benzimidazole; 1,4-Benzenedicarboxylic acid; 1,3,5-Benzenetricarboxylic acid; Coordination polymer; Crystal structure

1. Introduction

Coordination polymers have intriguing structural motifs and potential applications as functional materials [1, 2]. Much effort has been focused on the rational design and the controlled synthesis of coordination polymers based on transition metal ions and multidentate ligands. Research into coordination polymers assembled by polycarboxylate and *N*-heterocyclic ligands has led to numerous practical and conceptual developments in coordination polymers [3, 4]. Polycarboxylate ligands such as 1,4-benzenedicarboxylate, 1,3,5-benzenetricarboxylate and 1,2,4,5-benzenetetracarboxylate possess 2–4 carboxyl groups and exhibit varied coordination as well as the potential for multiple protonations [5–7]. This makes them very appealing for the design of coordination polymers.

There has been growing interest in structures and properties of coordination polymers constructed by *N*-heterocyclic ligands such as triazole, imidazole and their

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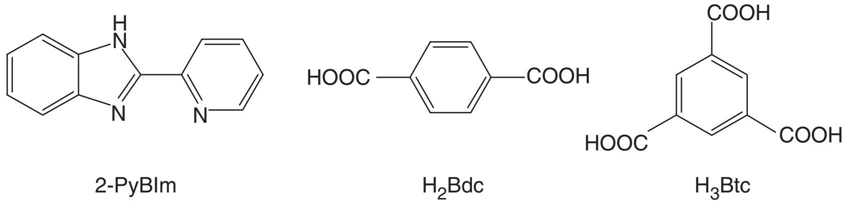
Journal of Coordination Chemistry
ISSN 0095-8972 print/ISSN 1029-0389 online © 2008 Taylor & Francis
DOI: 10.1080/00958970801958980



Scheme 1.

derivatives [8]. A number of transition metal 2-(*n*-pyridyl)benzimidazole (scheme 1) coordination polymers have been reported, wherein *n*-PyBIm ligands are deprotonated or neutral [9–11]. In the course of our investigation of coordination polymers, three polycarboxylate metal coordination polymers with 2-PyBIm have been prepared and structurally characterized. Herein, we report their syntheses, crystal structures, thermal and luminescent properties.

2. Experimental

2.1. *Materials and measurements*

All chemicals were of reagent grade and used without further purification. 2-(2-pyridyl)benzimidazole was prepared according to the literature method [12]. Elemental analyses (C, H, N) were performed on a Vario EL III elemental analyzer. Infrared spectra were recorded on a Nicolet A370 spectrometer with KBr pellets in the 400–4000 cm^{-1} region. TG-DSC analyses were completed on a Netzsch STA 449C thermal analyzer from 20 to 800°C at a heating rate of 10°C min^{-1} in air. Fluorescence spectra were recorded on a Shimadzu RF-5301 spectrophotometer.

2.2. *Synthesis*

$[Cu(Bdc)(2\text{-}PyBIm)]_n$ (1): A mixture of $Cu(NO_3)_2 \cdot 3H_2O$ (0.1 mmol), 1,4-benzenedicarboxylic acid (0.1 mmol), 2-(2-pyridyl)benzimidazole (0.1 mmol) and 8 mL H_2O was stirred for 30 min and then transferred into a 15-mL Teflon-lined reactor. The mixture was heated at 180°C for 72 h under autogenous pressure. Upon cooling to room temperature at a rate of 10°C h^{-1}, green crystals of **1** were collected in 35% yield based on Cu. Anal. Found: C, 55.66; H, 3.25; N, 9.94. Calcd for $C_{20}H_{13}CuN_3O_4$: C, 56.81; H, 3.10; N, 9.94. IR (KBr, cm^{-1}): 3219 m, 1597 s, 1560 s, 1453 m, 1393 s, 1373 s, 1353 s, 840 m, 754 s.

$[Cu(HBtc)(2\text{-}PyBIm)]_n$ (2): A mixture of $Cu(NO_3)_2 \cdot 3H_2O$ (0.2 mmol), 1,3,5-benzenetricarboxylic acid (0.1 mmol), 2-(2-pyridyl)benzimidazole (0.1 mmol) and 8 mL H_2O was stirred for 30 min and then transferred into a 15-mL Teflon-lined reactor. The mixture was heated at 170°C for 72 h under autogenous pressure. Upon cooling to room temperature at a rate of 10°C h^{-1}, blue crystals of **2** were collected in 40% yield based on Cu. Anal. Found: C, 53.61; H, 2.84; N, 8.87. Calcd for $C_{21}H_{13}CuN_3O_6$: C, 54.02; H, 2.81; N, 9.00. IR (KBr, cm^{-1}): 1715 m, 1613 s, 1586 m, 1447 m, 1360 s, 1260 m, 798 m, 746 m, 682 m.

[$Cd_2(HBtc)_2(2\text{-}PyBIm)_2]_n \cdot nH_2O$ (3): A mixture of $Cd(NO_3)_2 \cdot 4H_2O$ (0.2 mmol), 1,3,5-benzenetricarboxylic acid (0.1 mmol), 2-(2-pyridyl)benzimidazole (0.1 mmol) and 8 mL H_2O was stirred for 30 min and then transferred into a 15-mL Teflon-lined reactor. The mixture was heated at 160°C for 72 h under autogenous pressure. Upon cooling to room temperature at a rate of 10°C h^{-1}, yellow crystals of **3** were collected in 30% yield based on Cd. Anal. Found: C, 48.78; H, 2.93; N, 8.59. Calcd for $C_{42}H_{28}Cd_2N_6O_{13}$: C, 48.08; H, 2.67; N, 8.01. IR (KBr, cm^{-1}): 1691 s, 1612 s, 1548 s, 1438 s, 1365 s, 1296 m, 749 s, 697 m.

2.3. *Crystal structure determination*

Well-shaped single crystals of **1**–**3** were selected for X-ray diffraction. Data collections were performed with graphite-monochromatic Mo-Kα radiation ($\lambda = 0.71073$ Å) on a Bruker Smart Apex-II CCD diffractometer at $T = 293(2)$ K. Determinations of the crystal system, orientation matrix and cell dimensions were performed according to established procedures. Lorentz polarization and absorption corrections were applied. The structures were solved by direct methods with the SHELXS-97 program and refined by full-matrix least squares on F^2 with the SHELXL-97 program [13]. All non-hydrogen atoms were refined anisotropically, and hydrogen atoms were located and included at their calculated positions. The crystal data and structure refinement results are summarized in table 1. Selected bond lengths and angles are given in table 2.

Table 1. Crystallographic data and structure refinement for **1**–**3**.

	1	**2**	**3**
Empirical formula	$C_{20}H_{13}CuN_3O_4$	$C_{21}H_{13}CuN_3O_6$	$C_{42}H_{28}Cd_2N_6O_{13}$
Formula weight	422.87	466.88	1049.50
Crystal system	Monoclinic	Monoclinic	Triclinic
Space group	$P2_1/c$	$P2_1/c$	$P\bar{1}$
a (Å)	7.2848(8)	11.9289(12)	10.1151(12)
b (Å)	20.769(2)	10.438(1)	11.3086(14)
c (Å)	11.4072(12)	16.1295(16)	17.377(2)
α (°)	90	90	80.219(2)
β (°)	96.021(1)	108.594(1)	81.007(2)
γ (°)	90	90	83.283(2)
V (Å^3)	1716.4(3)	1903.5(3)	1926.4(4)
Z	4	4	2
D_c (Mg m^{-3})	1.636	1.629	1.809
Absorption coefficient (mm^{-1})	1.307	1.194	1.184
$F(000)$	860	948	1044
θ range for data collection (°)	2.05–25.00	2.36–25.05	2.03–25.05
Crystal size (mm^3)	0.30 × 0.30 × 0.20	0.20 × 0.20 × 0.10	0.30 × 0.20 × 0.20
Measured reflections	8802	9484	9987
Independent reflections	3020 [$R_{int} = 0.0301$]	3366 [$R_{int} = 0.0313$]	6682 [$R_{int} = 0.0273$]
Data/restraints/parameters	3020/0/253	3366/0/282	6682/0/578
Goodness-of fit on F^2	1.053	1.003	1.053
R [$I > 2\sigma(I)$]	$R_1 = 0.0353$, $wR_2 = 0.0774$	$R_1 = 0.0373$, $wR_2 = 0.0930$	$R_1 = 0.0460$, $wR_2 = 0.1082$
R (all data)	$R_1 = 0.0480$, $wR_2 = 0.0842$	$R_1 = 0.0503$, $wR_2 = 0.1002$	$R_1 = 0.0629$, $wR_2 = 0.1199$
Largest diff. peak and hole (e Å^{-3})	0.325 and −0.399	1.121 and −0.287	0.290 and −0.590

Table 2. Selected bond lengths (Å) and angles (°) for **1**–**3**.

Complex **1**			
Cu(1)–N(1)	2.043(2)	Cu(1)–O(3A)	2.0114(19)
Cu(1)–N(2)	1.987(2)	Cu(1)–O(4A)	2.343(2)
Cu(1)–O(1)	1.844(4)	Cu(1)–O(2)	2.012(2)
N(2)–Cu(1)–N(1)	81.11(10)	N(1)–Cu(1)–O(3A)	162.91(9)
O(1)–Cu(1)–O(2)	59.44(8)	N(2)–Cu(1)–O(2)	161.11(9)
O(3A)–Cu(1)–O(4A)	59.96(7)	O(1)–Cu(1)–O(4A)	158.80(8)
Complex **2**			
Cu(1)–O(1)	1.9417(19)	Cu(1)–N(1)	2.043(3)
Cu(1)–O(1A)	2.323(2)	Cu(1)–N(2)	1.971(3)
Cu(1)–O(6B)	1.967(2)		
O(1)–Cu(1)–N(2)	176.13(9)	O(1)–Cu(1)–O(1A)	79.19(8)
N(1)–Cu(1)–O(6B)	166.72(10)	Cu(1)–O(1)–Cu(1A)	100.81(8)
Complex **3**			
Cd(2)–O(6)	2.296(4)	Cd(1)–O(1)	2.245(4)
Cd(2)–O(7)	2.581(4)	Cd(1)–O(2)	2.521(4)
Cd(2)–O(8)	2.384(4)	Cd(1)–O(5)	2.417(4)
Cd(2)–O(11)	2.386(3)	Cd(1)–O(7)	2.338(4)
Cd(2)–O(12)	2.494(3)	Cd(1)–N(1)	2.370(5)
Cd(2)–N(4)	2.412(5)	Cd(1)–N(2)	2.282(4)
Cd(2)–N(5)	2.276(4)		
O(7)–Cd(2)–O(12)	172.69(12)	N(5)–Cd(2)–O(11)	138.46(13)
O(6)–Cd(2)–N(4)	159.28(15)	O(7)–Cd(1)–N(1)	154.31(13)
Cd(1)–O(7)–Cd(2)	102.89(13)	O(2)–Cd(1)–O(5)	134.09(12)

3. Results and discussion

3.1. *Synthesis and infrared spectra*

Complexes **1**–**3** have been prepared by hydrothermal reactions. 1,4-Benzenedicarboxylic acid and 1,3,5-benzenetricarboxylic acid are deprotonated under hydrothermal conditions and act as dianionic ligands. 2-(2-Pyridyl)benzimidazole remains neutral and forms 1 : 1 : 1 ternary complexes **1**–**3** with metal ions and Bdc^{2-} or $HBtc^{2-}$. These complexes have been characterized by IR spectra and elemental analyses.

In the IR spectrum of **1**, the characteristic carboxylate bands of $\nu_{as}(COO^-)$ and $\nu_s(COO^-)$ appear at 1560 and 1393 cm^{-1}, respectively. A splitting of 167 cm^{-1} (separation between ν_{as} and ν_s) is equal to that for ionic Na_2Bdc, indicating that the carboxylate groups of Bdc^{2-} coordinate Cu(II) in a chelating mode [14]. Complexes **2** and **3** contain $HBtc^{2-}$ and show $\nu_{as}(COOH)$ vibration absorptions at 1715 and 1691 cm^{-1}, respectively. For **2**, the strong absorption peaks at 1613 and 1360 cm^{-1} are assigned to $\nu_{as}(COO^-)$ and $\nu_s(COO^-)$ stretching vibrations of $HBtc^{2-}$. A splitting of 253 cm^{-1} indicates that the carboxylate groups coordinate Cu(II) monodentate or monodentate-bridging. For **3**, two carboxylate groups show different absorption bands. One carboxylate group has absorption peaks at 1612 (ν_{as}) and 1438 (ν_s) cm^{-1}, and the other carboxylate group shows 1548 (ν_{as}) and 1365 (ν_s) cm^{-1} absorptions. This indicates that the carboxylate groups coordinate Cd(II) in different modes. The X-ray structural analysis further reveals that one carboxylate group coordinates in a chelating mode, while the other carboxylate group coordinates in a bidentate-bridging mode.

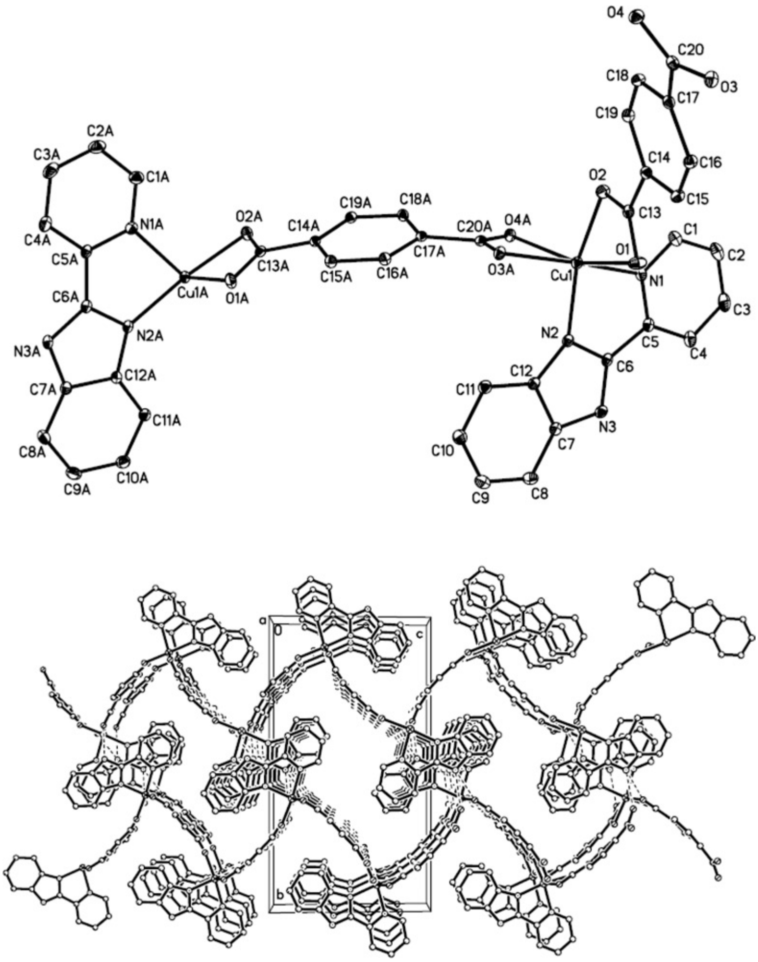


Figure 1. Asymmetric structural unit and packing diagram of **1**.

3.2. *Descriptions of the crystal structures*

X-ray structural analysis revealed that **1** is a one-dimensional zigzag chain coordination polymer. As shown in figure 1, the Cu(1) is in a distorted octahedral coordination environment coordinating two 2-PyBIm N atoms and four carboxyl O atoms. The equatorial plane is formed by coordination of N(1), N(2), O(2) and O(3A), while the axial positions are occupied by the O(1) and O(4A) atoms. The bond angles N(1)–Cu(1)–O(3A), N(2)–Cu(1)–O(2) and O(1)–Cu(1)–O(4A) are 162.91(9), 161.11(9), 158.80(8)°, respectively. 2-PyBIm is a chelating bidentate ligand towards Cu(1), with bond lengths of Cu(1)–N(1) 2.043(2) and Cu(1)–N(2) 1.987(2) Å. Both carboxyl groups coordinate Cu(1) in chelating bidentate mode. Bond lengths Cu(1)–O(1) and Cu(1)–O(2) are 1.844(4) and 2.012(2) Å, while Cu(1)–O(3A) and Cu(1)–O(4A) are 2.0114(19) and 2.343(2) Å, respectively.

In 2-PyBIm, the pyridyl ring is coplanar with the benzimidazole plane (dihedral angle 1.90°). The aromatic ring of 2-PyBIm is nearly perpendicular to the benzene ring of Bdc

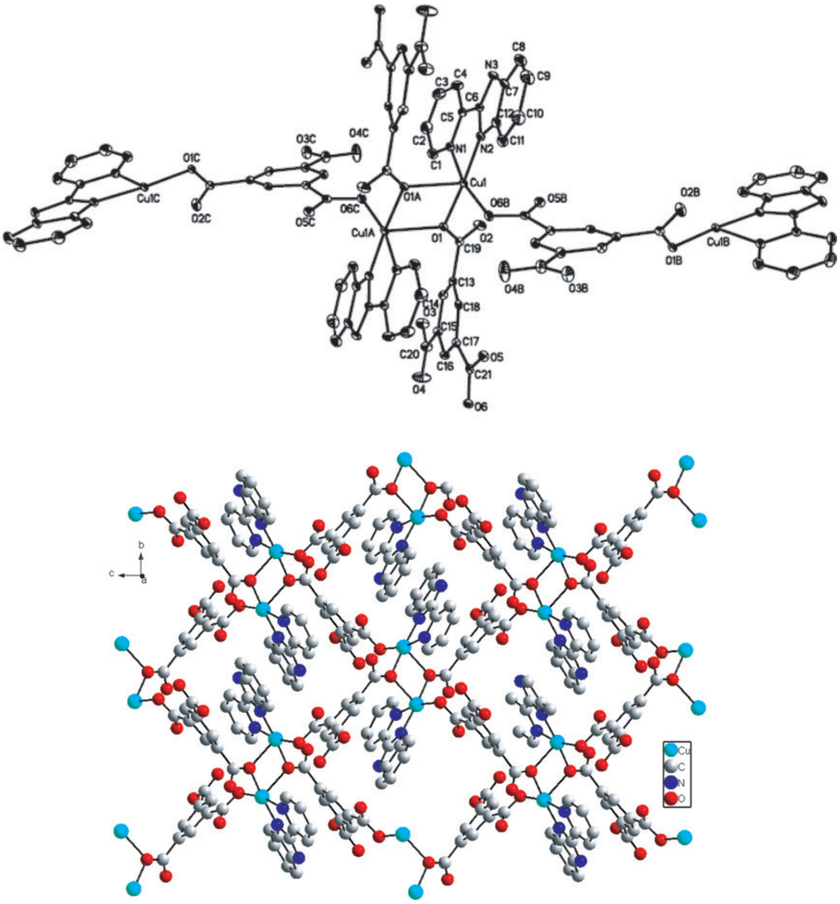


Figure 2. Asymmetric structural unit and two-dimensional (4,4) network of **2**.

with a dihedral angle of 96.8°. As the packing diagram shows, both carboxyl groups of Bdc are coordinated *cis* to copper, leading to formation of a one-dimensional zigzag chain polymeric structure. The zigzag chains are arranged in an ABAB parallel fashion, leading to some π–π stacking interaction between aromatic rings of 2-PyBIm with an average distance of 3.53 Å. There exist hydrogen bonding interactions between N(3)–H with O(1C) or O(4D). Such π–π stacking and H–bonding interactions assemble the one-dimensional zigzag chains of **1** into a three-dimensional supramolecular framework.

Complex **2** is a two-dimensional coordination polymer. As shown in figure 2, Cu(1) is square pyramidal. The basal plane is formed by O(1), N(1), N(2) and O(6B), while the axial site is occupied by O(1A). The bond angles for O(1)–Cu(1)–N(2), N(1)–Cu(1)–O(6B) and O(1A)–Cu(1)–O(1) are 176.13(9), 166.72(10) and 79.19(8)°, respectively. Similar to **1**, the 2-PyBIm is chelating bidentate to the copper with bond lengths of Cu(1)–N(1) 2.043(2) and Cu(1)–N(2) 1.971(3) Å. 1,3,5-Benzenetricarboxylate is a partially deprotonated tridentate ligand. One carboxylate of HBtc dianion coordinates monodentate to Cu(1) with a Cu(1)–O(6B) bond length of 1.967(2) Å. The other carboxylate is monodentate bridging linking Cu(1) and Cu(1A) using O(1).

The Cu(1)–O(1) and Cu(1A)–O(1) bond lengths are 1.9417(19) and 2.323(2) Å, respectively. All other bond lengths and angles are as expected [15].

Each HBtc coordinates to three coppers through two carboxyl groups. Two copper atoms are connected through two bridging oxygen atoms to form a rhombic Cu_2O_2 unit (Cu–O–Cu bond angle is 100.81°). As the packing diagram shows, **2** displays a complicated two-dimensional (4,4) network with nodes of Cu_2O_2 dimers and HBtc bridges. A Cu_2O_2 dimer with (4, 4) network structure was previously observed in the acetylenedicarboxylate complex $[Cu(C_4O_4)(bipy)]_n$ [16].

Complex **3** has a three-dimensional framework in which 1,3,5-benzenetricarboxylic acid is partially deprotonated. As shown in figure 3, **3** possesses two unique Cd(II) sites. Cd(1) is six-coordinate with two 2-PyBIm N atoms and four carboxyl O atoms, while Cd(2) is seven-coordinate by two 2-PyBIm N atoms and five carboxyl O atoms. 2-PyBIm is a neutral, chelating, bidentate ligand. There are two unique HBtc dianions in the asymmetric structural unit, one acts as a tetradentate ligand and the other as a pentadentate ligand. Cd(1) and Cd(2) are linked by one bridging carboxyl group and one bridging oxygen. The distance between Cd(II) atoms is 3.850 Å. The HBtc dianion-connected Cd(2-PyBIm) units use both carboxylate groups to form a three-dimensional polymeric framework. There exist strong hydrogen bonding interactions between carboxyl group, amino group and lattice water.

The 2-(2-pyridyl)benzimidazole units can be neutral bidentate ligands and anionic tridentate ligands once the benzimidazole deprotonated [17, 18]. In **1**–**3**, 2-PyBIm is a neutral, chelating, bidentate ligand. Attempts to prepare coordination polymers featuring tridentate 2-PyBIm ligands were unsuccessful.

3.3. *Thermal stability and luminescence*

Thermal analyses of **1**–**3** were performed in the temperature range 20–800°C. Complex **1** is stable up to 300°C without weight loss, but decomposed rapidly from 300 to 360°C with 37.97% weight loss corresponding to release of 1,4-benzenedicarboxylate (Calcd: 35.02%). 2-PyBIm was released in the range 370–630°C with the final 18.01% CuO residue (Calcd: 18.81%). Complex **2** is stable up to 200°C and decomposed by two steps in the 200–250 and 330–370°C regions with total weight loss of 44.90%. This process corresponds to decomposition of 1,3,5-benzenetricarboxylate (Calcd: 41.16%) and indicates that carboxyl groups may be released in two steps [19]. 2-PyBIm was released in the range 380–620°C (found: 38.59%; Calcd: 41.81%), with the final 16.51% CuO residue (Calcd: 17.04%). Complex **3** lost 2.62% lattice water in 80–100°C (Calcd: 1.72%); 1,3,5-benzenetricarboxylate decomposed in the range 300–410°C with 37.59% weight loss (Calcd: 36.61%); 2-PyBIm was released at 420–800°C with 36.73% weight loss (Calcd: 37.20%).

The excitation and emission spectra of **3** in the solid state at room temperature are shown in figure 4. Photoexcitation of **3** with light of 374 nm gives strong emission with maximum at 454 nm. This emission band might be attributed to ligand-to-metal charge transfer (LMCT), as observed in other polynuclear Cd(II) complexes [20], and suggests that **3** is a good candidate as a blue-light emitter.

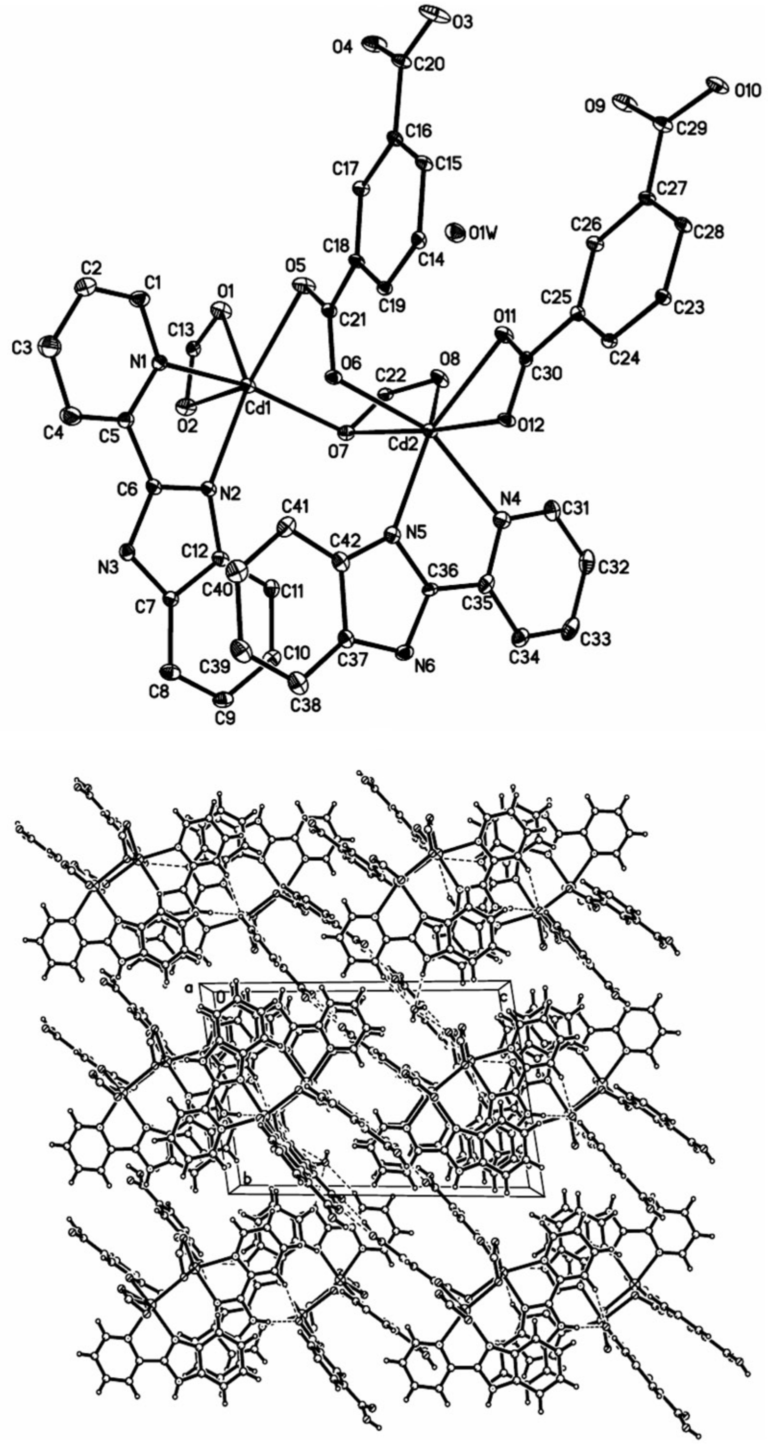


Figure 3. Asymmetric structural unit and packing diagram of **3**.

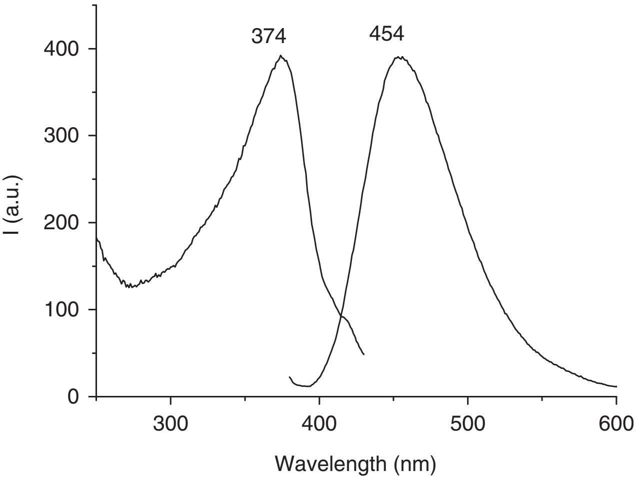


Figure 4. Fluorescence spectrum of **3**, left half is excitation spectrum and right half is emission spectrum.

Supplementary material

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Center, CCDC-647552 for **1**, CCDC-647553 for **2** and CCDC-651073 for **3**. Copies of the data can be obtained free of charge from CCDC, 12 Union Road, Cambridge, CB2IEZ, UK (Email: deposit@ccdc.cam.ac.uk).

Acknowledgements

We thank the Research Foundation for Returned Chinese Scholars from Overseas of the Chinese Education Ministry (7A14219) and the Development Foundation of the Shanghai Education Commission (03AK35).

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