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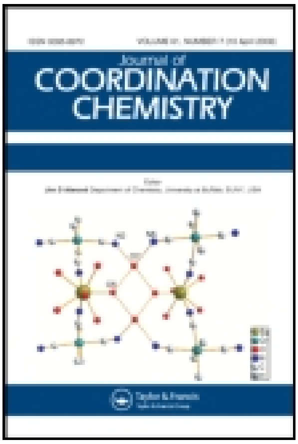

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Syntheses, structures, and fluorescence of two cadmium compounds $[Cd_2(pqc)_4(phen)_2(H_2O)_2]\cdot 2H_2O$ and $\{[Cd(pqc)_2(bpy)(H_2O)_2]\cdot 2H_2O\}_n$

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Syntheses, structures, and fluorescence of two cadmium compounds $[Cd_2(pqc)_4(phen)_2(H_2O)_2]\cdot 2H_2O$ and $\{[Cd(pqc)_2(bpy)(H_2O)_2]\cdot 2H_2O\}_n$

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Assembly of 2-phenylquinoline-4-carboxylic acid and $Cd(CH_3COO)_2\cdot 2H_2O$ or $CdSO_4\cdot 8H_2O$ gave two new complexes $[Cd_2(pqc)_4(phen)_2(H_2O)_2]\cdot 2H_2O$ (**1**) and $\{[Cd(pqc)_2(bpy)(H_2O)_2]\cdot 2H_2O\}_n$ (**2**) (pqcH = 2-phenylquinoline-4-carboxylic acid, phen = 1,10-phenanthroline, bpy = 4,4′-bpy). Complexes **1** and **2** have been characterized by single-crystal X-ray diffraction, IR, powder X-ray diffraction, thermogravimetric analyses, and fluorescence spectra. Structural analyses reveal that **1** is binuclear; the binuclear Cd(II) units are linked by intermolecular hydrogen bonds and π–π interactions to generate a supramolecular structure. Complex **2** possesses a 1-D infinite chain with chains further assembled into a 3-D supramolecular network by π–π, H-bonding, and C–H $\cdots\pi$ interactions.

Keywords: Cadmium compounds; Crystal structures; Fluorescence

1. Introduction

Coordination compounds with novel structural motifs and potential applications in photoluminescence, catalysis, sensor, and electronics [1–3] have been reported, of which the structures or properties have been successfully attempered by controlling the inorganic/organic components, such as the sizes/shapes of functional groups, coordination geometries of metal ions, and flexibility/symmetry of the organic ligands [4–6]. However, there are still many challenges involving preparation of complexes with directed dimensional networks, exhibiting distinct physical or chemical properties. A key step to assemble structures is to select appropriate ligands with coordination functional groups. Previous studies have proved that rigid multidentate ligands with N/O donors are good choices [7, 8]. 2-Phenylquinoline-4-carboxylic acid (pqcH) possesses interesting characteristics: (a) it has a carboxylate and a pyridine nitrogen, which are apt to form strong bonding to metal ions; (b) it has a large conjugated unit, which can have π-electron transitions resulting in photoluminescence emission.

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A few coordination compounds of 2-phenylquinoline-4-carboxylates have been assembled [9–21], but cadmium(II) complexes with 2-phenylquinoline-4-carboxylates have not been reported. Herein, we report two new compounds constructed from Cd(II) and pqc^-, characterized by IR, thermogravimetric analyses (TGA), fluorescence, powder, and single-crystal X-ray diffraction.

2. Experimental

2.1. *Materials and physical measurements*

All commercially available chemicals were of reagent grade and used as received. FT-IR spectra were recorded from 4000 to 400 cm^{-1} in KBr pellets on a Shimadzu FTIR-8900 spectrometer. Fluorescence measurements were obtained on a LS 50B Luminescence Spectrometer (Perkin-Elmer, Inc., USA). TGA were carried out on a STA449C integration thermal analyzer in air with a heating rate of 10°C min^{-1}. Powder X-ray diffraction (PXRD) data were collected on a Bruker D8 Focus X-ray diffractometer using Cu-Kα radiation. The calculated PXRD patterns were produced using the *SHELXTL-XPOW* programs.

2.1.1. Synthesis of $[Cd_2(pqc)_4(phen)_2(H_2O)_2]\cdot 2H_2O$ (1). 2-Phenylquinoline-4-carboxylic acid (0.0120 g, 0.05 mmol), $Cd(CH_3COO)_2\cdot 2H_2O$ (0.0159 g, 0.05 mmol), and 1,10-phen (0.0098 g, 0.05 mmol) in EtOH/H_2O solution (1 : 1, 10 mL) were placed in a 25 mL stainless steel reactor fitted with a Teflon liner, heated to 100°C for 3 days, and then cooled to room temperature. Colorless block-like crystals were obtained and dried in air. Yield: 76% (based on Cd). Anal. Calcd (%) for $C_{88}H_{64}Cd_2N_8O_{12}$: C, 64.04; H, 3.91; N, 6.79. Found (%): C, 63.87; H, 3.84; N, 7.36. IR (KBr, cm^{-1}): 3438(s), 3059(w), 1573(s), 1547(m), 1518 (m), 1494(w), 1445(w), 1429(w), 1387(vs), 1371(m), 1319(s), 1234(m), 1154(w), 1101(w), 1078 (w), 1028(m), 912(w), 865(w), 842(s), 813(s), 770 (vs), 725(s), 698(s), 652(s), 567(w), 517(w).

2.1.2. Synthesis of $\{[Cd(pqc)_2(bpy)(H_2O)_2]\cdot 2H_2O\}_n$ (2). 2-Phenylquinoline-4-carboxylic acid (0.0240 g, 0.10 mmol), $CdSO_4\cdot 8H_2O$ (0.0774 g, 0.10 mmol), 4,4′-bpy (0.0198 g, 0.10 mmol), and KOH (0.0056 g, 0.10 mmol) in H_2O solution (10 mL) were placed in a 25 mL stainless steel reactor fitted with a Teflon liner, heated to 100°C for 2 days, and then cooled to room temperature. Colorless block-like crystals were obtained and dried in air. Yield: 85% (based on Cd). Anal. Calcd (%) for $C_{88}H_{64}Cd_2N_8O_{12}$: C, 60.26; H, 4.33; N, 6.69. Found (%): C, 61.04; H, 4.48; N, 6.73. IR (KBr, cm^{-1}): 3381(m), 3120(m), 3062(m), 1600(s), 1562(m), 1545(w), 1491(s), 1415(s), 1392(s), 1318(vs), 1222(vs), 1149(m), 1107(m), 1043(w), 964(m), 893(m), 851(m), 806(vs), 769(vs), 730(s), 697(s), 653 (s), 628(s), 604(vs), 569(m), 517(w), 492(vs).

2.2. *X-ray crystallography*

A high quality crystal of the complex was mounted on a glass fiber. Data were collected on a BRUKER APEX II CCD diffractometer equipped with graphite-monochromated

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

Compound	**1**	**2**
Empirical formula	$C_{88}H_{64}Cd_2N_8O_{12}$	$C_{42}H_{36}CdN_4O_8$
Formula weight	1650.27	837.15
Temperature (K)	298(2)	298(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Triclinic
Space group	*P2₁/c*	*P-1*
Unit cell dimensions (Å, °)		
a	14.8706(2)	11.7397(13)
b	13.8406(2)	11.7658(13)
c	17.8687(3)	15.2204(17)
α	90	75.4580(10)
β	96.034(2)	70.9740(10)
γ	90	69.2060(10)
Volume (Å^3), Z	3657.32(9), 2	1836.1(4), 2
Calculated density (mg m^{-3})	1.499	1.514
Absorption coefficient (mm^{-1})	0.654	0.656
F(000)	1680	856
Crystal size (mm^3)	0.32 × 0.29 × 0.09	0.28 × 0.19 × 0.12
θ range for data collection (°)	3.70–25.00	1.87–27.54
Limiting indices	$-17 \le h \le 17$; $-9 \le k \le 16$; $-21 \le l \le 21$	$-15 \le h \le 15$; $-15 \le k \le 14$; $-16 \le l \le 19$
Reflections collected/unique	22,103/6407 [R(int) = 0.0305]	16,055/8269 [R(int) = 0.0323]
Completeness to theta = 32.27	99.5%	97.6%
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	6407/3/624	8269/12/528
Goodness-of-fit on F^2	0.920	1.031
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0276$, $wR_2 = 0.0591$	$R_1 = 0.0396$, $wR_2 = 0.0769$
R indices (all data)	$R_1 = 0.0410$, $wR_2 = 0.0611$	$R_1 = 0.0658$, $wR_2 = 0.0916$

Mo-Kα radiation ($\lambda = 0.71073$ Å) using an ω scan mode at 298(2) K. Empirical absorption corrections were applied using SADABS [22]. The structure was solved by direct methods and refined with a full-matrix least-squares technique using SHELXTL-97 [23]. Anisotropic thermal parameters were applied to all non-hydrogen atoms. Hydrogens were generated geometrically. Crystallographic data and details of data collection and refinement for **1** and **2** are listed in table 1. Selected bond lengths and angles are given in table 2.

3. Results and discussion

3.1. *Crystal structure of* $[Cd_2(pqc)_4(phen)_2(H_2O)_2]\cdot 2H_2O$ *(1)*

Compound **1** is stable in air and insoluble in common solvents. Single-crystal X-ray diffraction analysis indicates that **1** is a binuclear structure and crystallizes in the monoclinic, space group *P2₁/c* with $Z = 2$. The asymmetric unit consists of two pqc$^-$ of which one is monodentate and the other bidentate bridging, one 1,10-phen, one cadmium, one coordinated water molecule, and one guest water molecule. The bidentate bridging pqc$^-$ connects two Cd(II) by carboxylates, forming the binuclear structure. As shown in figure 1, Cd1A is symmetrically related by Cd1 (symmetry code: $-x$, $-y+1$, $-z+1$) and the two Cd(II) ions possess the same

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

Compound **1**		Compound **2**	
Cd(1)–O(1)	2.1995(17)	Cd(1)–O(3)	2.311(2)
Cd(1)–O(3)	2.2779(17)	Cd(1)–O(1)	2.317(2)
Cd(1)–O(2)#1	2.3132(16)	Cd(1)–O(6)	2.322(3)
Cd(1)–O(5)	2.3771(18)	Cd(1)–N(4)#1	2.335(2)
Cd(1)–N(4)	2.3331(19)	Cd(1)–O(5)	2.339(2)
Cd(1)–N(3)	2.3432(19)	Cd(1)–N(3)	2.358(2)
O(2)–Cd(1)#1	2.3132(16)	O(3)–Cd(1)–O(1)	169.09(8)
O(1)–Cd(1)–O(3)	88.40(7)	O(3)–Cd(1)–O(6)	91.45(10)
O(1)–Cd(1)–O(2)#1	93.07(6)	O(1)–Cd(1)–O(6)	94.10(10)
O(3)–Cd(1)–O(2)#1	93.07(6)	O(3)–Cd(1)–N(4)#1	92.76(9)
O(1)–Cd(1)–N(4)	109.40(8)	O(1)–Cd(1)–N(4)#1	96.88(9)
O(3)–Cd(1)–N(4)	161.91(7)	O(6)–Cd(1)–N(4)#1	87.16(10)
O(2)#1–Cd(1)–N(4)	89.31(6)	O(3)–Cd(1)–O(5)	91.05(9)
O(1)–Cd(1)–N(3)	173.37(6)	O(1)–Cd(1)–O(5)	83.87(9)
O(3)–Cd(1)–N(3)	90.28(7)	O(6)–Cd(1)–O(5)	176.34(10)
O(2)#1–Cd(1)–N(3)	93.49(6)	N(4)#1–Cd(1)–O(5)	90.05(9)
N(4)–Cd(1)–N(3)	71.66(7)	O(3)–Cd(1)–N(3)	87.37(9)
O(1)–Cd(1)–O(5)	80.17(7)	O(1)–Cd(1)–N(3)	83.36(8)
O(3)–Cd(1)–O(5)	94.37(7)	O(6)–Cd(1)–N(3)	88.90(10)
O(2)#1–Cd(1)–O(5)	169.79(7)	N(4)#1–Cd(1)–N(3)	176.05(10)
N(4)–Cd(1)–O(5)	85.78(7)	O(5)–Cd(1)–N(3)	93.89(9)

Symmetry code: #1 $-x, -y+1, -z+1$ for **1**; #1 $x-1, y, z$ for **2**.

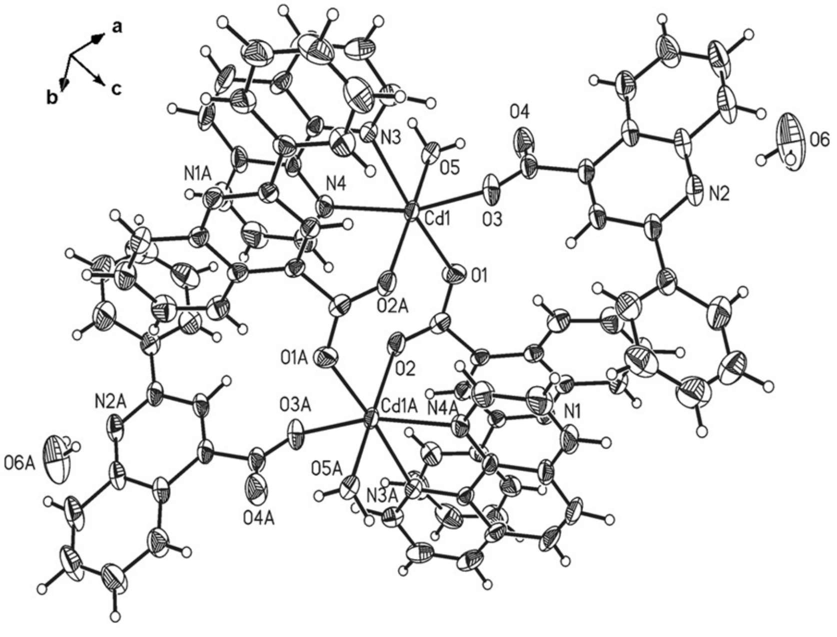

Figure 1. Binuclear structure of **1**.

coordination environment. Cd(II) is a slightly distorted octahedron formed by two nitrogens of phenanthroline and four oxygens, three from different pqc$^-$ and one from coordinated water, with Cd–O distances ranging from 2.199 to 2.377 Å and Cd–N distances of 2.343 and 2.333 Å.

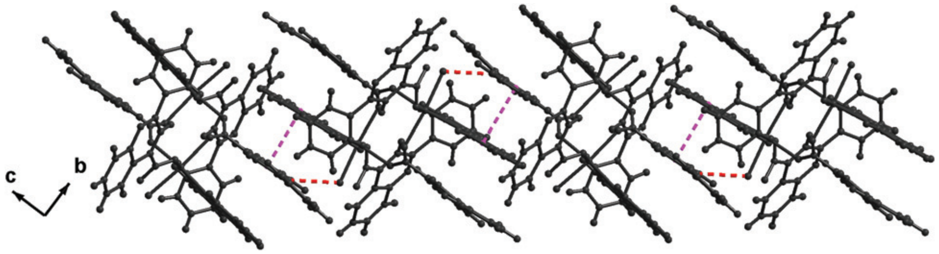

Figure 2. View of hydrogen-bonding and π–π interaction of **1**.

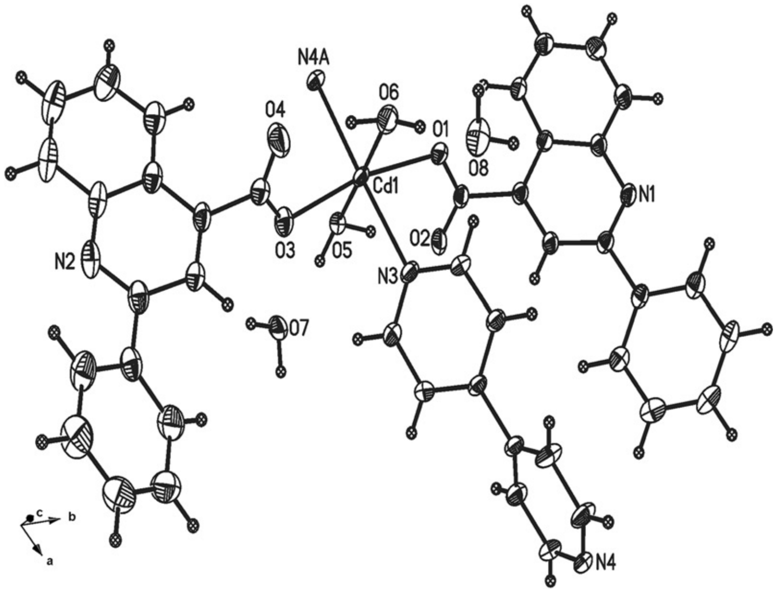

Figure 3. The local coordination environment of Cd(II) for **2**.

In **1**, the centroid-to-centroid separation of the nearest two benzene rings of pqc^- (N1, C8, C3, C2, C10, C9) and phen ligand (N3, C33, C34, C35, C36, C44) is 3.672 Å, which implies strong intermolecular π–π stacking interaction. The dinuclear Cd(II) units are linked by intermolecular hydrogen bonding (O5–H5B ··· N1 = 2.855 Å, symmetry code: x, $-y+1/2$, $z-1/2$) and π–π interaction to generate a 1-D chain (figure 2); the chains are further assembled into a supramolecular structure by intermolecular interactions.

3.2. *Crystal structure of* $\{[Cd(pqc)_2(bpy)(H_2O)_2]\cdot 2H_2O\}_n$ *(2)*

Compound **2** consists of 1-D chains $\{[Cd(pqc)_2(bpy)(H_2O)_2]\cdot 2H_2O\}_n$. The basic structural unit of **2** is shown in figure 3. The central Cd^{2+} in **2** is six-coordinate with two nitrogens from different 4,4′-bpy ligands, two oxygens from pqc^-, and two oxygens from two coordinated water molecules. Two oxygens are axial and the other four

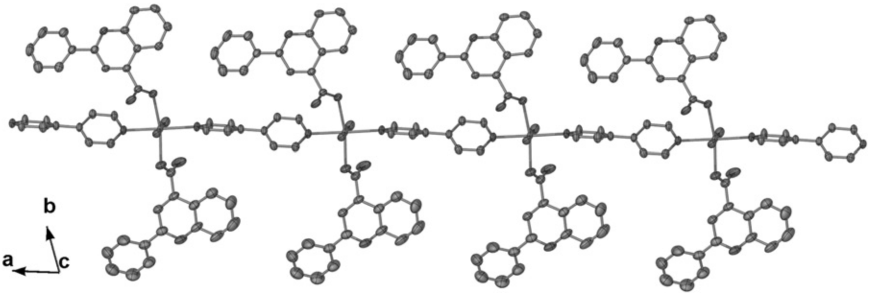

Figure 4. 1-D chain structure of **2**.

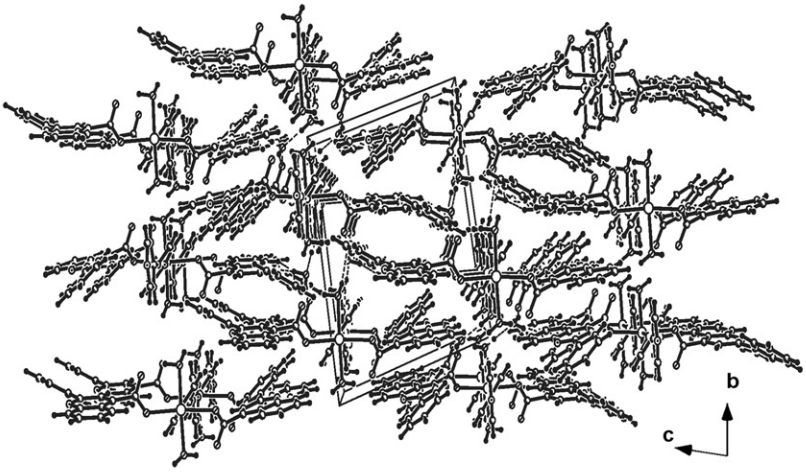

Figure 5. 3-D structure in the *bc* plane for **2**.

equatorial with Cd–O distances ranging from 2.311 to 2.339 Å and Cd–N distances being 2.335 and 2.358 Å, respectively. Therefore, cadmium is a slightly distorted octahedral geometry. The 4,4′-bpy bridges two symmetry related metal centers. The distance of the two adjacent Cd(II) is 11.979 Å, and the Cd ··· Cd ··· Cd angle formed by three adjacent cadmiums is 180.00°, as the crystallographic coordinates for the Cd ion are refined it is possible to calculate the error in Cd–Cd distance and Cd–Cd–Cd angle in **2**. Each 4,4′-bpy bridges two cadmiums to generate a 1-D infinite chain (figure 4), the coordination style is similar to the previous work [24, 25]. The chains are further extended into an infinite 3-D network coordination polymer *via* π–π interactions and C–H ··· π interactions and H-bonding interactions (figure 5).

3.3. *PXRD analyses*

To confirm the homogeneity of the material synthesized under the solvo(hydro)thermal conditions, the experimental/simulated PXRD analyses of **1** and **2** were collected.

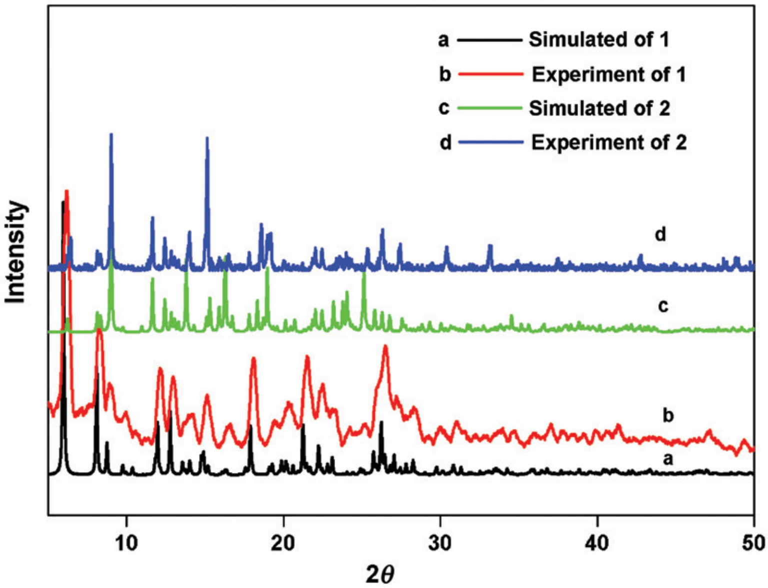

Figure 6. Experimental (*b* for **1** and *d* for **2**) and simulated (*a* for **1** and *c* for **2**) powder XRD patterns of **1** and **2**.

As depicted in figure 6, the experimental pattern resembles the simulated single crystal data, indicating that the bulk products obtained are homogeneous in nature.

3.4. *Thermogravimetric analyses*

TGA experiments were performed on crystalline samples of **1** and **2** from 40°C to 700°C to determine the thermal stabilities, an important aspect for metal–organic frameworks [26, 27]. For **1**, the first weight loss of 3.27% (Calcd: 3.32%) corresponds to three water molecules per formula unit $[Cd_2(pqc)_4(phen)_2(H_2O)_2]\cdot 2H_2O$ from 80 to 140°C, implying removal of the guest water molecule and part of the coordinated water molecule. A sharp drop in weight is observed after 280°C, indicating decomposition of the framework of the complex. In **2**, the weight loss of 8.68% (Calcd: 8.60%) from 122°C to 312°C corresponds to loss of four water molecules per formula unit $\{[Cd(pqc)_2(bpy)(H_2O)_2]\cdot 2H_2O\}_n$; then a sharp weight loss with increasing temperature (figure 7) indicates decomposition of the framework.

3.5. *Fluorescence and UV-Vis spectra*

The solid-state photoluminescence spectra of **1** and **2** and the ligands have been measured at room temperature. As shown in figure 8, the ligands exhibit strong emission with maximum wavelength centered at *ca* 420 nm for free pqcH, 363 and 380 nm for free phen and 360 nm for free 4,4′-bipyridine, which originate from the ligand-to-ligand π_L–π_L* charge transfer (LLCT). In the solid state, **1** displays intense fluorescent emission at 400 nm upon photoexcitation at 308 nm, while **2** displays intense

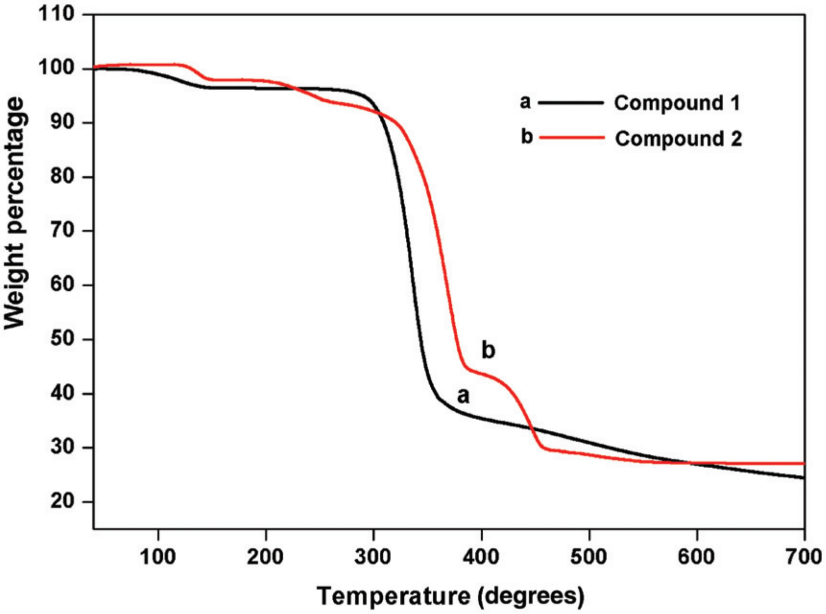

Figure 7. TGA curve of the title **1** and **2**.

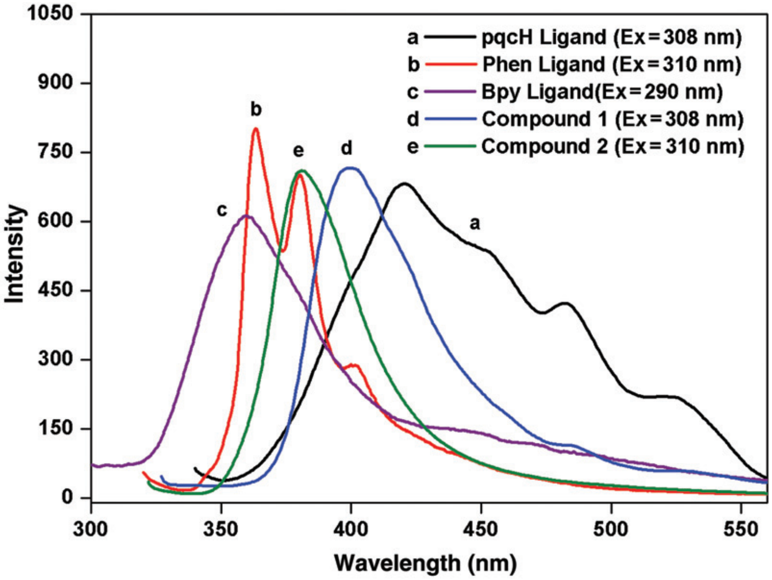

Figure 8. Solid-state emission spectra of the free organic ligand, **1** and **2**.

fluorescent emission at 380 nm upon photoexcitation at 310 nm. The strong maximum emission peak (400 nm) of **1** may originate from the LLCT transition emission of the pqc and phen ligands. The strong emission peak (380 nm) of **2** may originate from the LLCT transition emission of the 4,4′-bpy and pqc ligands. More detailed theoretical

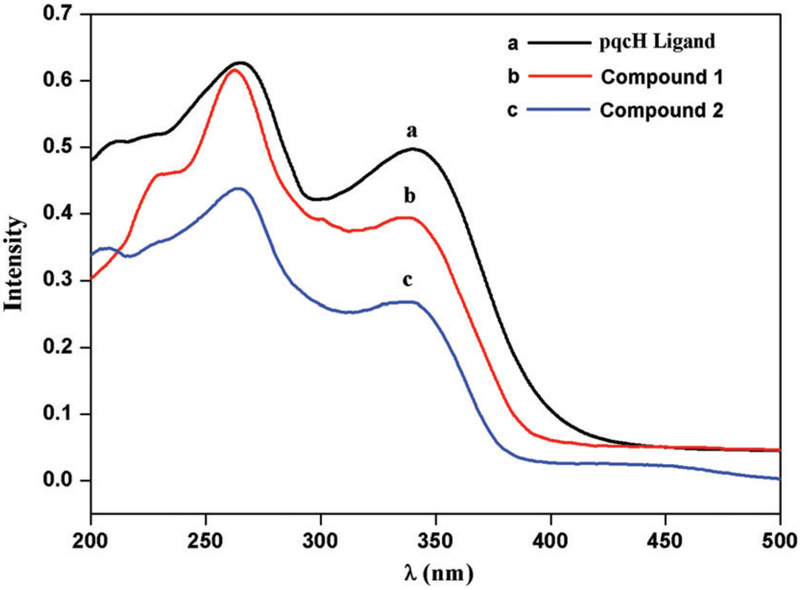

Figure 9. UV-Vis spectra of pqcH ligand and **1** and **2**.

and spectroscopic studies are in progress for a better understanding of the luminescent mechanism [28–30].

The UV-Vis spectra of **1**, **2**, and pqc$^-$ at room temperature are illustrated in figure 9. The ligand exhibits high-energy bands at 270 and 350 nm due to π–π* transitions of pqcH. The slightly less intense absorptions of **1** and **2** at 270 and 350 nm are tentatively ascribed to metal-perturbed π–π* transitions in pqc$^-$, probably mixed with some d(Cd)-π* metal-to-ligand charge transfer (MLCT) character [31].

Supplementary material

Crystallographic data (excluding structure factors) for the complex in this article have been deposited at the Cambridge Crystallographic Data Center as supplementary publication (CCDC-780024 for **1** and 791279 for **2**). Copies of the data can be obtained free of charge from the Cambridge Crystallographic Data Center, 2 Union Road, Cambridge CB2 1EZ, UK; Fax: (+44)-1223-336033; E-mail: deposit@ccdc.cam.ac.uk; www: www.ccdc.cam.ac.uk

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