

# Crystal Structures and Luminescent Properties of Two Zinc(II) Complexes with 2-Phenylquinoline-4-carboxylates

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Assembly of 2-phenylquinoline-4-carboxylic acid and  $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$  gives rise to two complexes  $[\text{Zn}_2(\text{C}_{16}\text{H}_{10}\text{NO}_2)_4(\text{phen})_2(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}$  (**I**) and  $[\text{Zn}_2(\text{C}_{16}\text{H}_{10}\text{NO}_2)_4(\text{bpy})_2]_n$  (**II**) ( $\text{phen} = 1,10\text{-phenanthroline}$ ,  $\text{bpy} = 4,4'\text{-bipyridine}$ ). Complexes **I** and **II** have been characterized by x-ray single-crystal diffraction, infrared (IR), powder x-ray diffraction (PXRD), thermogravimetric analysis (TGA), and fluorescence spectra. Structural analysis reveals that **I** is binuclear in structure; two Zn(II) centers are bridged by two carboxylate groups and the terminal water molecule occupies a axial position of the octahedral geometry, which further extends the binuclear unit into a supramolecular framework via intermolecular hydrogen bonding and  $\pi \cdots \pi$  interactions. Complex **II** possesses a one-dimensional infinite zigzag chain architecture by bridging of the 4,4'-bipyridine.

**Keywords** crystal structures, fluorescence, 2-phenylquinoline-4-carboxylates, zinc complexes

## INTRODUCTION

Rapid development of metal-organic frameworks has been made in recent years not only for their potential applications in materials science but also for fascinating architectures and topologies.<sup>[1,2]</sup> Hydrogen bonding and  $\pi$ - $\pi$  stacking interactions are vital to design and construct supramolecular compounds.<sup>[3-6]</sup> Meanwhile, assistant ligands such as 1,10-phenanthroline and 4,4'-bipyridine may contribute to the various architectures of the supramolecular compounds. How to control the molecular structures and topologies in the process of self-assembly has attracted extensive attention. It has been proved that the rigid ligands containing nitrogen and/or

carboxylate oxygen donor atoms are fine building motifs for the construction of metal complexes. 2-Phenylquinoline-4-carboxylic acid (H<sub>pqc</sub>), aside from its significance in biological systems,<sup>[7,8]</sup> also possesses fascinating coordination behaviors, such as asymmetric geometry and multiple coordination sites, which have been widely used to design and synthesize metal-organic coordination complexes because of the carboxylate group and/or pyridine nitrogen atom.<sup>[9-15]</sup> Zinc is a flexible transition metal in adopting various coordination geometries in its complexes. In this article, we report the synthesis and characterization of compound **I** and compound **II**. Compound **I** is binuclear structure, and further extends its units to form a supramolecular framework via intermolecular hydrogen bonding interactions (O-H $\cdots$ O) and  $\pi \cdots \pi$  interactions. Compound **II** has a one-dimensional (1D) chain formed by bridging of 4,4'-bipyridine.

## EXPERIMENTAL

### Materials and Measurements

All commercially available chemicals were of reagent grade and used as received without further purification. The Fourier-transform infrared (FT-IR) spectra were recorded from KBr pellets in the range of 4000–400  $\text{cm}^{-1}$  on a Shimadzu FTIR-8900 spectrometer. All fluorescence measurements were carried out on an LS 50B Luminescence Spectrometer (Perkin-Elmer, Inc., USA). Thermogravimetric analyses (TGA) were carried out in an air atmosphere with a heating rate of 10°C/min on a STA449C integration thermal analyzer. Powder x-ray diffraction (PXRD) data were collected on a Bruker D8 Focus x-ray diffractometer using Cu  $K\alpha$  radiation. The calculated PXRD patterns were produced using the SHELXTL-XPOW program.

### Synthesis of the Complex

#### Preparation of $[\text{Zn}_2(\text{C}_{16}\text{H}_{10}\text{NO}_2)_4(\text{phen})_2(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}$ (**I**)

2-Phenylquinoline-4-carboxylic acid (0.0123 g, 0.05 mmol),  $\text{Zn}(\text{OAc})_2 \cdot 2\text{H}_2\text{O}$  (0.0210 g, 0.10 mmol), phen (0.0198 g, 0.10 mmol), and KOH (0.0028 g, 0.05 mmol) in  $\text{H}_2\text{O}$  solution (10 mL) were placed in a 25-mL stainless-steel reactor fitted with a Teflon liner and heated to 100°C for 2 days, then cooled to room temperature; colorless block-like crystals of (**I**) were

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TABLE 1  
Crystal data and structure refinement for **I** and **II**

Complex	(I)	(II)
Empirical formula	C <sub>88</sub> H <sub>64</sub> N <sub>8</sub> O <sub>12</sub> Zn <sub>2</sub>	C <sub>84</sub> H <sub>56</sub> N <sub>8</sub> O <sub>8</sub> Zn <sub>2</sub>
Formula weight	1556.21	1436.11
Temperature (K)	296(2)	296(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Triclinic
Space group	P2 <sub>1</sub> /c	P-1
a (Å)	14.8487(17)	12.9829(12)
b (Å)	13.8206(16)	14.2101(14)
c (Å)	17.507(2)	19.9969(19)
α (°)	90	91.3570(10)
β (°)	97.5650(10)	106.5730(10)
γ (°)	90	101.9530(10)
V (Å <sup>3</sup> )	3561.4(7)	3445.8(6)
Z	2	2
Crystal size (mm)	0.35 × 0.15 × 0.12	0.45 × 0.22 × 0.15
Density (calculated) (mg/m <sup>3</sup> )	1.451	1.384
Absorption coefficient (mm <sup>-1</sup> )	0.748	0.763
F(000)	1608	1480
Theta range for data collection (°)	1.88 to 27.54°	1.47 to 27.47°
Limiting indices	-19 ≤ h ≤ 19, -17 ≤ k ≤ 16, -22 ≤ l ≤ 22	-16 ≤ h ≤ 16, -18 ≤ k ≤ 18, -25 ≤ l ≤ 21
Reflections collected	30737	30364
Independent reflections	8209 [R(int) = 0.0625]	15453 [R(int) = 0.0383]
Completeness to theta	27.54/99.9%	27.47/98.0%
Refinement method	Full-matrix least-squares on F <sup>2</sup>	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	8209 / 0 / 502	15453 / 0 / 916
Goodness of fit on F <sup>2</sup>	1.008	1.012
Final R indices [I > 2σ(I)]	R1 = 0.0378, wR2 = 0.0974	R1 = 0.0479, wR2 = 0.1270
R indices (all data)	R1 = 0.0573, wR2 = 0.1090	R1 = 0.0904, wR2 = 0.1502
Largest diff. peak and hole (e.Å <sup>-3</sup> )	0.341 and -0.473	0.573 and -0.477

obtained. The crystals were collected and dried in air. Yield: 74% (based on Zn). Anal.: Calcd. (%) for C<sub>88</sub>H<sub>64</sub>N<sub>8</sub>O<sub>12</sub>Zn<sub>2</sub>: C, 67.91; H, 4.15; N, 7.20. Found (%): C, 67.88; H, 4.23; N, 7.35. IR (KBr, cm<sup>-1</sup>): 3428(vs), 3060(w), 1746(w), 1624(vs), 1574(vs), 1546(vs), 1519(m), 1425(m), 1389(vs), 1319(m), 841(w), 813(w), 770(vs), 724 (m), 698(m), 651(m).

#### Preparation of [Zn<sub>2</sub>(C<sub>16</sub>H<sub>10</sub>NO<sub>2</sub>)<sub>4</sub>(4,4'-bpy)<sub>2</sub>]<sub>n</sub> (**II**)

2-Phenylquinoline-4-carboxylic acid (0.0123 g, 0.05 mmol), Zn(OAc)<sub>2</sub>·2H<sub>2</sub>O (0.0105 g, 0.05 mmol), 4,4'-bpy (0.0098 g, 0.05 mmol), and KOH (0.0028 g, 0.05 mmol) in H<sub>2</sub>O solution (10 mL) were placed in a 25-mL stainless reactor fitted with a Teflon liner and heated to 100°C for 2 days, then cooled to room temperature; colorless block-like crystals of (**II**) were obtained. The crystals were collected and dried in air. Yield: 67% (based on Zn). Anal.: Calcd. (%) for C<sub>84</sub>H<sub>56</sub>N<sub>8</sub>O<sub>8</sub>Zn<sub>2</sub>: C, 70.25; H, 3.93; N, 7.80. Found (%): C, 70.14; H, 3.90; N, 3.83. IR (KBr, cm<sup>-1</sup>): 3423(vs), 3059(w), 2925(w), 2362(w), 1620 (vs), 1569(m), 1542(m), 1419(m), 1389(vs), 1317(m), 1221(m), 1112(s), 1024(m), 813(m), 774(s), 699(m), 619(m), 566(w), 511(w).

#### DATA COLLECTION, STRUCTURAL DETERMINATION, AND REFINEMENT

##### Crystal Structure Determination

A suitable crystal with dimensions of 0.35 mm × 0.15 mm × 0.12 mm for **I** and 0.45 mm × 0.22 mm × 0.15 mm for **II** were carefully selected under a polarizing microscope and glued at the tip of a thin glass fiber with cyanoacrylate adhesive. Single-crystal determination was performed on a Bruker Apex II CCD diffractometer equipped with graphite-monochromatized Mo-Kα radiation (λ = 0.71073 Å) for data collection by using an ω-scan mode at room temperature. The structures were solved by a direct method<sup>[16]</sup> and refined with SHELXTL-97 by full-matrix least-squares technique on F<sup>2</sup>. All nonhydrogen atoms were refined anisotropically, and the hydrogen atoms were included in the final refinement on the calculated positions bonded to their carrier atoms.<sup>[17]</sup> The crystallographic data and other pertinent information of complexes **I** and **II** are summarized in Table 1, and the selected bond lengths and bond angles are given in Table 2. Hydrogen bond lengths and bond angles are given in Table 3 for **I**. Crystallographic data for complex **I** and **II** were deposited at the Cambridge Crystallographic

TABLE 2  
Bond lengths (Å) and bond angles (°) for **I** and **II**

Compound <b>I</b>			
Bond lengths			
Zn(1)–O(1)#1	2.0348(14)	Zn(1)–N(3)	2.1541(16)
Zn(1)–O(4)	2.1238(14)	Zn(1)–O(3)	2.1685(14)
Zn(1)–O(2)	2.1295(14)	Zn(1)–N(2)	2.1741(16)
Bond angles			
O(1)#1–Zn(1)–O(4)	90.88(6)	O(2)–Zn(1)–O(3)	173.58(5)
O(1)#1–Zn(1)–O(2)	91.73(5)	N(3)–Zn(1)–O(3)	85.78(6)
O(4)–Zn(1)–O(2)	90.73(5)	O(1)#1–Zn(1)–N(2)	176.61(6)
O(1)#1–Zn(1)–N(3)	101.51(7)	O(4)–Zn(1)–N(2)	90.46(6)
O(4)–Zn(1)–N(3)	167.47(6)	O(2)–Zn(1)–N(2)	91.36(6)
O(2)–Zn(1)–N(3)	90.73(6)	N(3)–Zn(1)–N(2)	77.06(6)
O(1)#1–Zn(1)–O(3)	83.71(6)	O(3)–Zn(1)–N(2)	93.10(6)
#1 $-x + 2, -y + 1, -z$			
Compound <b>II</b>			
Bond lengths			
Zn(1)–O(1)	1.932(3)	Zn(1)–N(7)	2.093(2)
Zn(1)–N(5)	2.056(2)	Zn(1)–O(3)	2.272(4)
Zn(1)–O(4)	2.090(3)	O(5)–Zn(2)	2.160(3)
O(6)–Zn(2)	2.204(3)	O(7)–Zn(2)	2.114(2)
O(8)–Zn(2)	2.271(2)	N(6)–Zn(2)	2.090(2)
N(8)–Zn(2)#1	2.109(2)	Zn(2)–N(8)#2	2.109(2)
Bond angles			
O(1)–Zn(1)–N(5)	117.07(11)	N(6)–Zn(2)–O(5)	92.11(10)
O(1)–Zn(1)–O(4)	104.07(13)	N(8)#2–Zn(2)–O(5)	157.07(11)
N(5)–Zn(1)–O(4)	128.03(14)	O(7)–Zn(2)–O(5)	103.02(10)
O(1)–Zn(1)–N(7)	116.10(12)	N(6)–Zn(2)–O(6)	99.26(9)
N(5)–Zn(1)–N(7)	98.06(9)	N(8)#2–Zn(2)–O(6)	98.36(10)
O(4)–Zn(1)–N(7)	90.41(11)	O(7)–Zn(2)–O(6)	157.74(10)
O(1)–Zn(1)–O(3)	92.68(15)	O(5)–Zn(2)–O(6)	59.77(10)
N(5)–Zn(1)–O(3)	90.05(12)	N(6)–Zn(2)–O(8)	155.26(9)
O(4)–Zn(1)–O(3)	56.27(14)	N(8)#2–Zn(2)–O(8)	87.00(9)
N(7)–Zn(1)–O(3)	141.38(14)	O(7)–Zn(2)–O(8)	60.21(8)
N(6)–Zn(2)–N(8)#2	98.72(9)	O(5)–Zn(2)–O(8)	91.57(9)
N(6)–Zn(2)–O(7)	95.15(9)	O(6)–Zn(2)–O(8)	103.71(9)
#1 $x + 1, y + 1, z$		#2 $x - 1, y - 1, z$	

Data Center (CCDC-795198 for **I** and CCDC-795199 for **II**).

## RESULTS AND DISCUSSION

### Synthesis

Various kinds of structures of metal-organic complexes may be isolated by employing different synthetic techniques. The pH value, temperature, and mole ratio are very important to obtain new complexes. Compound **I** and **II** were obtained in base environment through hydrothermal reaction. When KOH was removed, or the temperature was raised to 140 °C, compounds **I** and **II** could not be obtained and only white deposition was

obtained. The reaction of 2-phenylquinoline-4-carboxylic acid, Zn(OAC)<sub>2</sub>·2H<sub>2</sub>O, and phen in a mole ratio of 1/2/2 gave rise to compound **I**, and the reaction of 2-phenylquinoline-4-carboxylic acid, Zn(OAC)<sub>2</sub>·2H<sub>2</sub>O, and 4,4'-bpy in a mole ratio of 1/1/1 gave rise to compound **II**. When the mole ratio was changed, we could not obtain high-quality crystals.

### Structure Description of Complex **I**

As shown in Figure 1, complex **I** is a binuclear structure, in which Zn1A is a symmetrical component related by Zn1 (symmetry code:  $-x + 2, -y + 1, -z$ ), and the two Zn(II) ions possess the same coordination environment. The zinc ion adopts a distorted octahedral coordinated geometry formed by two N

TABLE 3  
Hydrogen bond lengths (Å) and bond angles (°) for **I**

Donor-H	d(D-H)	d(H...A)	d(D...A)	∠DHA	Symmetry code
O(3)-H(1)	0.966	1.902	2.862	171.91	$N(1)[-x + 2, y + 1/2, -z + 1/2]$
O(6)-H(2)	0.984	2.238	3.167	157.14	$O(4)[-x + 1, -y + 1, -z]$
O(6)-H(2)	0.984	2.537	3.186	123.39	$O(2)[-x + 1, -y + 1, -z]$

atoms and four oxygen atoms, of which three are from different 2-phenylquinoline-4-carboxylic acid ligands and one from coordinated water, with Zn-O distances ranging from 2.035 to 2.168 Å and Zn-N distance being 2.154 Å and 2.174 Å.

In complex **I**, the centroid-to-centroid separation of the nearest two benzene rings of pqc<sup>-</sup> ligands and 1,10-phen is 3.581 Å, which implies an intermolecular  $\pi$ - $\pi$  stacking interaction. The binuclear units are linked by intermolecular hydrogen bonding (O3-H1...N1 = 2.862 Å, symmetry code:  $-x + 2, y + 1/2, -z + 1/2$ ) and  $\pi$ - $\pi$  interaction to generate a one-dimensional chain

(Figure 2). The chain is further assembled into a supramolecular framework by the other intermolecular hydrogen bonding (O6-H2...O4 = 3.167 Å, symmetry code:  $-x + 1, -y + 1, -z$ ; O6-H2...O2 = 3.186 Å, symmetry code:  $-x + 1, -y + 1, -z$ ).

### Structure Description of Complex II

As shown in Figure 3, the asymmetry unit of compound **II** consists of two Zn(II) ions together with two 4,4'-bpy and four 2-phenylquinoline-4-carboxylate ligands. The Zn1 is coordinated by three carboxyl oxygen atoms from two different

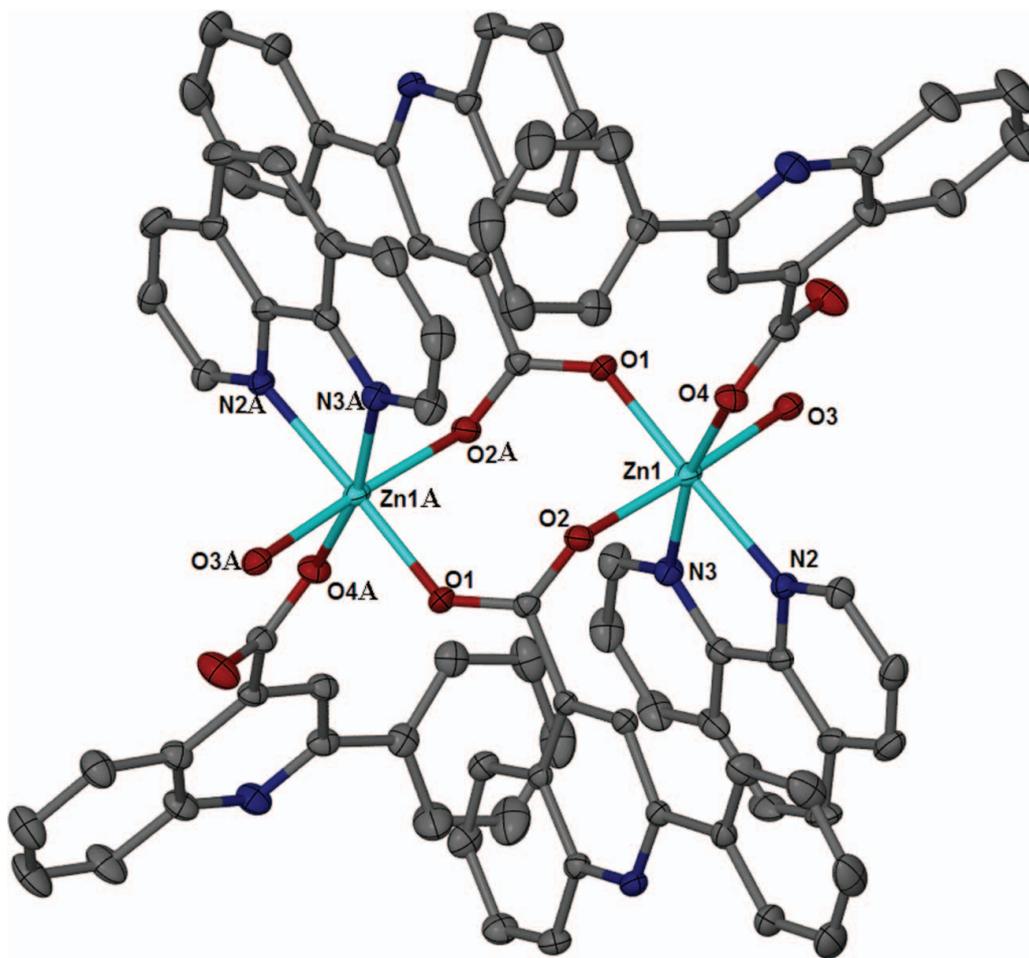


FIG. 1. Binuclear structure of compound **I**. Ellipsoids correspond to 30% probability. Hydrogen atoms and guest water are omitted for clarity. (Symmetry codes: A:  $-x + 2, -y + 1, -z$ ) (color figure available online.)

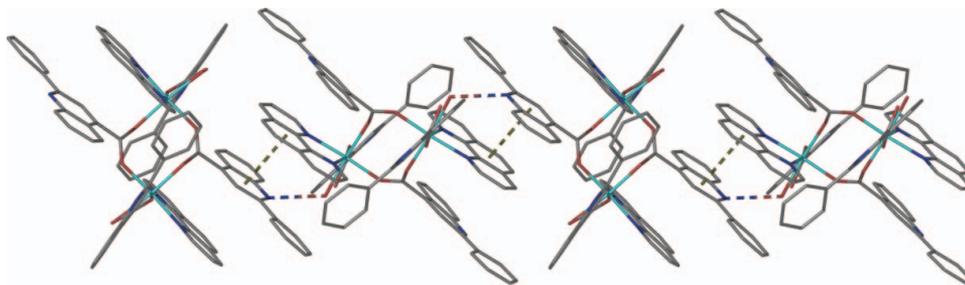


FIG. 2. View of 1D chain with hydrogen bonding and  $\pi$ - $\pi$  interaction of compound **I** (color figure available online.)

2-phenylquinoline-4-carboxylate ligands (O1, O3, O4) and two nitrogen atoms from two different 4,4'-bpy ligands (N5, N7), forming a distorted pyramid. The Zn2 is coordinated by four carboxyl oxygen atoms from two different 2-phenylquinoline-4-carboxylate ligands (O5, O6, O7, O8) and two nitrogen atoms from two different 4,4'-bpy ligands, forming a distorted octahedral geometry. In the complex **II**, Zn-O distances range from 1.932 (3) to 2.272 (4) Å and Zn-N distances from 2.056(2) to 2.109(2) Å. The asymmetry units of compound **II** are bridged by 4,4'-bpy, giving rise to a zigzag zinc chain (Figure 4).

As is well known, the bridging ligands are propitious to the information of polymeric structures, and the terminal ligands are in favor of the construction of multinuclear clusters with the metal ions. Therefore, the 4,4'-bpy ligands link Cd(II) ions to

form the one dimensional structure in compound **II** and compound **I** is the binuclear cluster by the carboxylate bridge and phen chelate to Cd(II) ions, in which the different structures are understandable in comparison with some previous reports in terms of coordination chemistry.<sup>[13,15]</sup>

### Power X-Ray Diffraction Analysis

In an attempt to confirm the homogeneity of the material synthesized under the hydrothermal condition, we have analyzed the PXRD pattern of **I** and **II** and correlated the results with the simulated powder pattern obtained from the single crystal data. As depicted in Figure 5, the basic features of the PXRD patterns resemble the simulated from single-crystal data for **I** and **II**, indicating that the bulk products obtained are homogeneous in nature.

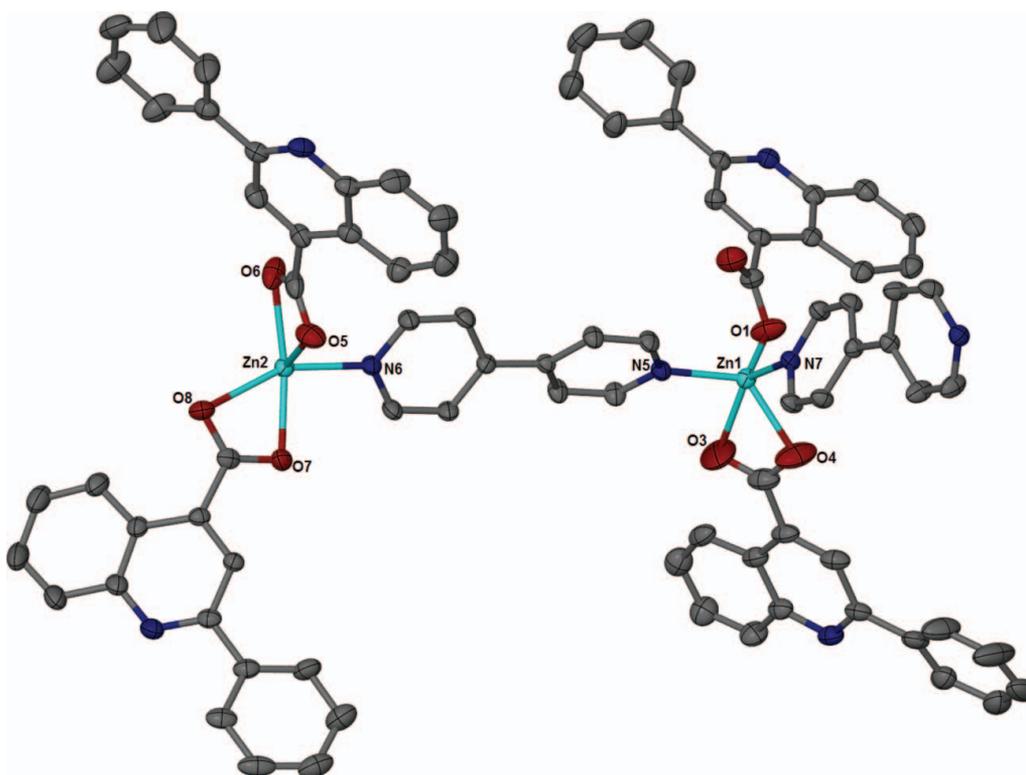


FIG. 3. Structure unit of compound **II**. Ellipsoids correspond to 30% probability. Hydrogen atoms are omitted for clarity (color figure available online.)

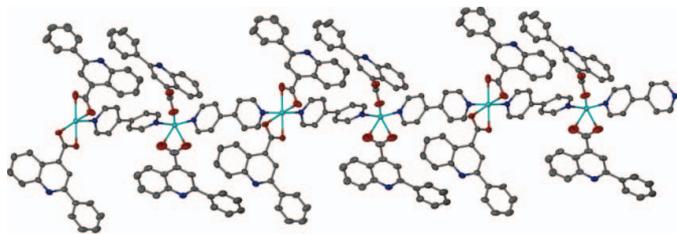


FIG. 4. View of the 1D chain of compound **II** (color figure available online.)

### IR Spectrum

The IR spectra of complexes **I** and **II** are very similar; the characteristic bands of carboxylate groups are shown in the range  $1560\text{--}1620\text{ cm}^{-1}$  for an asymmetric stretching and  $1370\text{--}1490\text{ cm}^{-1}$  for a symmetric stretching. The absorptions in the range  $1500\text{--}1420\text{ cm}^{-1}$  are attribute to characteristic peaks of the  $\text{C}=\text{N}$  and  $\text{C}=\text{C}$  bonds of the aromatic ring. The broad bands at  $\sim 3300\text{ cm}^{-1}$  correspond to the vibration of the water molecules in the complexes.

### Thermogravimetric Analyses

Thermogravimetric analysis (TGA) experiments were conducted to determine the thermal stability of the title complexes, which was an important aspect for metal-organic frameworks.<sup>[18,19]</sup> TGA was performed on crystalline samples of **I** and **II** under an air atmosphere from 40 to  $800^\circ\text{C}$ . In complex **I**, the first weight loss of 2.5% (calcd. 2.3%) corresponds to two guest water molecules per formula unit  $[\text{Zn}_2(\text{C}_{16}\text{H}_{10}\text{NO}_2)_4(\text{phen})_2(\text{H}_2\text{O})_2]\cdot 2\text{H}_2\text{O}$  from 100 to  $170^\circ\text{C}$ , implying removal of the guest water. Then a sharp drop of weight was observed after  $283^\circ\text{C}$ , indicating the decomposition of the framework of complex **I**. In compound **II**, the first weight loss of 65.1% (calcd: 69.1%) corresponds to the four  $\text{pqc}^-$  ( $\text{C}_{16}\text{H}_{10}\text{NO}_2$ ) molecules per formula unit

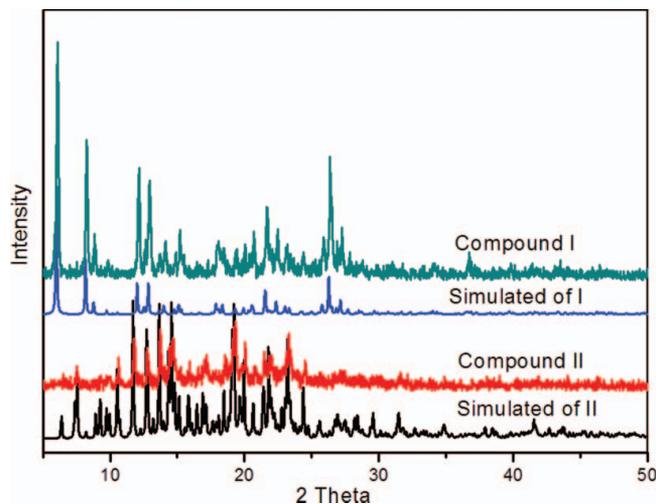


FIG. 5. Experimental and simulated powder XRD patterns of **I** and **II** (color figure available online.)

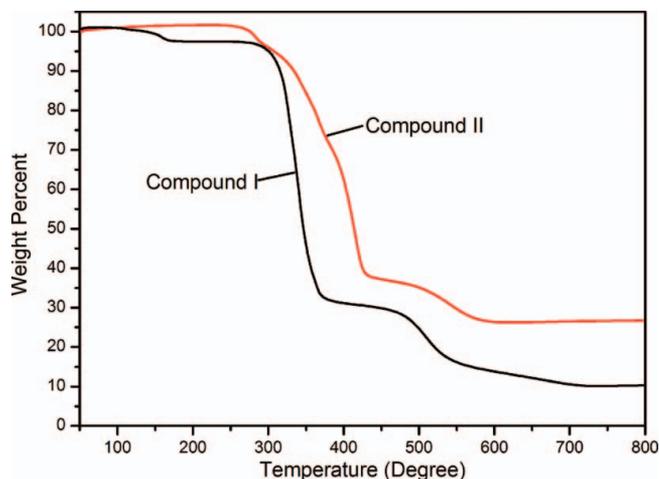


FIG. 6. TGA curve of the title complex **I** and **II** (color figure available online.)

$[\text{Zn}_2(\text{C}_{16}\text{H}_{10}\text{NO}_2)_4(4,4'\text{-bpy})_2]_n$  from 260 to  $500^\circ\text{C}$ , indicating the stability of the framework of the complex up to  $260^\circ\text{C}$  (Figure 6).

### Fluorescent Emission

Previous studies have shown that compounds containing zinc ions could exhibit fine photoluminescent properties.<sup>[20,21]</sup> Hence, we also investigated the photoluminescent properties of complexes **I** and **II** (Figure 7). In the solid state, strong photoluminescence emission bands at 390 and 430 nm are observed for complexes **I** and **II**, respectively, upon photoexcitation at 300 nm. The free 2-phenylquinoline-4-carboxylic acid exhibits photoluminescence emission at 420 nm, which may originate from the ligand-to-ligand  $\pi_L\text{--}\pi_L^*$  charge transfer (LLCT) transition emission. The position blue-shift (comparing with  $\text{pqc}^-$ ) and the red-shift (comparing with phen) of the strong emission peak (390 nm) and the fluorescent enhancement of compound

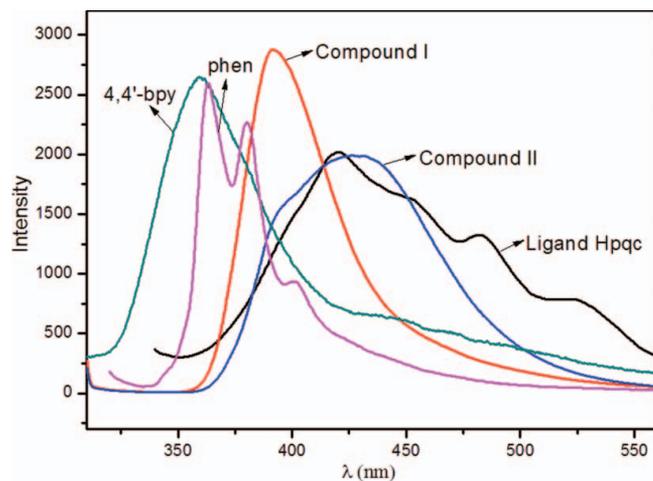


FIG. 7. Solid-state emission spectra of **I** and **II** at room temperature (color figure available online.)

**I** may be assigned to the intermolecular  $\pi$ - $\pi$  stacking interaction of phen and/or pqc<sup>-</sup> ligands or the effect of ligand-to-metal or metal-to-ligand charge transfer (LMCT or MLCT) transition emission similar to previous reports.<sup>[22]</sup> The emission bands at 430 nm of **II** could be attributed to the pqc<sup>-</sup> ligand donation from the coordination interactions or crystal packing interactions in the solid by radiationless decay of the intra-ligands.

## CONCLUSIONS

Two new zinc complexes with 2-phenylquinoline-4-carboxylates have been synthesized and characterized. TGA analyses show high thermal stabilities of the frameworks for **I** and **II**; the luminescence measurement suggests an intense strong emission at solid state. This work may provide useful information for employing rigid carboxylate ligands to assemble new functional materials.

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