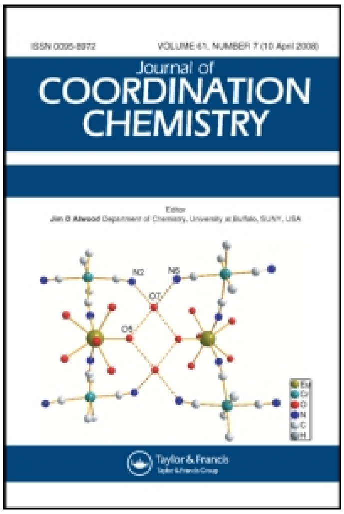

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Dinuclear Zn(II) and Hg(II) complexes of an angular dipyridyl ligand: syntheses, crystal structures, and properties

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Dinuclear Zn(II) and Hg(II) complexes of an angular dipyridyl ligand: syntheses, crystal structures, and properties

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The angular polytopic dipyridyl ligand 2,6-*bis*(quinoline-2-carboxamido)pyridine (H_2L) was prepared. Assemblies of H_2L with $ZnAc_2$ and $HgAc_2$ resulted in two new dinuclear complexes $[Zn_2(L)(Ac)_2]\cdot 1.5H_2O\cdot 0.5CH_3OH$ (**1**) and $[Hg_2(L)(Ac)_2]\cdot 5H_2O\cdot CH_3OH$ (**2**) where the doubly deprotonated L^{2-} bi-chelate as μ-$kN,N':kN'',N'''$, bridging the two metal centers (Ac = acetate). In **1**, the two Zn(II) ions are also doubly bridged by two Ac ions in a μ-kO : kO′ coordination, and thus each metal center adopts a distorted tetrahedral geometry. In **2**, each Ac ion is only terminal to Hg(II), in a rare distorted triangular or T-shaped coordination geometry. Free H_2L, **1**, and **2** emit interesting bluish-green fluorescence with strong intensities. Thermogravimetric analysis of **1** shows that the dinuclear structure of **1** is stable to 382°C.

Keywords: Angular dipyridyl ligand; Crystal structure; Dinuclear complex; Fluorescence property

1. Introduction

Carboxamido-containing ligands have been of increasing interest due to their various ligating behaviors to fabricate metal–organic architectures through coordination, hydrogen-bonding and π–π stacking interactions giving metal–organic assemblies with potential applications in asymmetric catalysis, dendrimer synthesis, molecular receptors, fluorescent materials, and so on [1–7]. Various ditopic and polytopic ligands with various coordination pockets of amide and azomethine groups can control the number and array of metal ions, and thus have the potential to yield homo- and heteropolynuclear complexes with special interest in multimetallic enzymes, catalytic reactions, and luminescent materials [8–12]. In order to efficiently prepare functional molecular materials, our group has synthesized *bis*(amidopyridine) ligands and their metal coordination complexes [13–17]. As a continuation, carboxamido-containing polytopic ligand 2,6-*bis*(quinoline-2-carboxamido)pyridine (H_2L) was designed and synthesized (scheme 1). The molecule has multiple hydrogen-bonding and coordination sites, and its two large carboxamido-containing *N*,*N*-bidentate chelating pockets are linked into the

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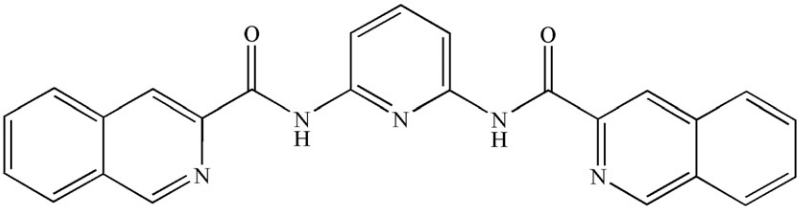

Scheme 1. Schematic representation of free ligand H_2L with C_{2v} symmetry.

2,6-diaminopyridyl backbone with an angular configuration. Thus, the organic spacer is an intriguing ligand in supramolecular chemistry. Herein, we report the syntheses, structures, and properties of two new dinuclear complexes $[Zn_2(L)(Ac)_2] \cdot 1.5H_2O \cdot 0.5CH_3OH$ (**1**) and $[Hg_2(L)(Ac)_2] \cdot 5H_2O \cdot CH_3OH$ (**2**) [Ac = acetate].

2. Experimental

2.1. *Materials and physical measurements*

Reagents and solvents were of analytical pure grades. Pyridine was dried and distilled from sodium hydroxide. Infrared (IR) spectra were recorded on a Bruker Vector22 spectrophotometer with KBr pellets from 400 to 4000 cm^{-1}. ^{1}H-NMR spectrum was recorded on a Varian Inova-500 spectrophotometer at room temperature and the chemical shifts are quoted in δ (ppm) relative to the deuterated solvent. Elemental analyses were performed with a Carlo-Erba 1106 elemental analyzer. Fluorescence spectra were determined in a solid state at room temperature on a Hitachi F-4500 fluorophotometer. Thermal analysis curve was scanned in a range of 35–700°C in air on a STA 409 PC thermal analyzer.

2.2. *Syntheses*

2.2.1. Synthesis of 2,6-*bis*(quinoline-2-carboxamido)pyridine (H_2L). 2,6-*Bis*(quinoline-2-carboxamido)pyridine was synthesized according to the literature [6] and recrystallized from DMF giving white crystals of H_2L (Yield: 33%; m.p.: >270°C). Selected IR (KBr, cm^{-1}): 3350(m), 1696(s), 1587(m), 1456(m), 1125(m), 946(m), and 773(m). ^{1}H-NMR (400 MHz, $CDCl_3$) $\delta = 10.70$ [s, 2H, NH] and 7.69–8.42 [m, py, and Ar, 15H].

2.2.2. Syntheses of $[Zn_2(L)(Ac)_2] \cdot 1.5H_2O \cdot 0.5CH_3OH$ (1) and $[Hg_2(L)(Ac)_2] \cdot 5H_2O \cdot CH_3OH$ (2). To a solution of $ZnAc_2 \cdot 2H_2O$ (0.0220 g, 0.1 mmol) in CH_3OH (15 mL), a solution of 2,6-*bis*(quinoline-2-carboxamido)pyridine (0.0210 g, 0.05 mmol) in $CHCl_3$ (15 mL) was added dropwise with stirring. After filtration, the filtrate was allowed undisturbed evaporation at ambient temperature. One week later, yellow crystals suitable for X-ray single crystal diffraction analysis were collected, washed with methanol, and dried in air, 0.033 g (Yield: 45%). Elemental Anal. Calcd (%) for

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

	1	**2**
Empirical formula	$C_{29.5}H_{26}N_5O_8Zn_2$	$C_{30}H_{35}Hg_2N_5O_{12}$
Formula weight	709.29	1058.81
Temperature (K)	296(2)	296(2)
Crystal system	Monoclinic	Triclinic
Space group	$C2/c$	$P\bar{1}$
Unit cell dimensions (Å, °)		
a	24.921(4)	7.814(2)
b	11.4938(18)	14.518(5)
c	23.300(4)	18.748(6)
α	90	107.479(5)
β	113.207(3)	98.829(5)
γ	90	103.466(5)
Volume (Å^3), Z	6133.8(17), 8	1914.8(11), 2
Calculated density (Mg m^{-3})	1.536	1.836
Crystal size (mm^3)	0.27 × 0.24 × 0.11	0.27 × 0.18 × 0.15
Goodness-of-fit on F^2	1.008	1.017
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0455$, $wR_2 = 0.1217$	$R_1 = 0.0520$, $wR_2 = 0.1251$
Largest difference peak and hole (e Å^{-3})	0.756 and −0.420	1.943 and −1.654

$C_{29.5}H_{26}N_5O_8Zn_2$: C 49.95, H 3.69, and N 9.87; found: C 49.68, H 3.70, and N 9.90. Selected IR (KBr, cm^{-1}): 3419(m), 1650(s), 1598(m), 1439(m), 1381(m), 1142(m), 944(m), and 767(m). Following the same synthesis as for **1**, crystals of **2** were obtained by replacing $ZnAc_2 \cdot 2H_2O$ with $HgAc_2 \cdot 2H_2O$ (Yield: 65%). Elemental Anal. Calcd (%) for $C_{30}H_{35}Hg_2N_5O_{12}$: C 34.03, H 3.33, and N 6.61; found: C 34.01, H 3.26, and N 6.56. Selected IR (KBr, cm^{-1}): 3475(m), 1620(s), 1597(m), 1431(m), 1360(m), 1149(m), 935(m), and 771(m).

2.3. *X-ray structural determination*

Crystallographic data for **1** and **2** were collected at 296(2) K on a Bruker Apex-II area-detector diffractometer with Mo-Kα radiation ($\lambda = 0.71073$ Å). Absorption corrections were applied using SADABS. The structures were solved by direct methods and refined with full-matrix least-squares on F^2 using the SHELXTL program package [18]. All non-hydrogen atoms were refined anisotropically. All hydrogens were assigned with common isotropic displacement factors and included in the final refinement using geometrical restraint. Crystal data are summarized in detail in table 1. Selected bond lengths and angles are listed in table 2.

3. Results and discussion

3.1. *Synthesis and IR spectra*

2,6-*Bis*(quinoline-2-carboxamido)pyridine (H_2L) was readily prepared through reaction of quinoline-2-carbonyl chloride with 2,6-diaminopyridyl in a 2 : 1 ratio and characterized by IR and ^{1}H-NMR. In the IR spectrum of H_2L, the strong and sharp band at

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

1			
Zn(1)–O(3)	1.935(3)	Zn(1)–O(1)	1.947(3)
Zn(1)–N(2)	1.938(3)	Zn(1)–N(1)	2.102(3)
Zn(2)–O(4)	1.943(3)	Zn(2)–O(2)	1.941(3)
Zn(2)–N(4)	1.942(3)	Zn(2)–N(5)	2.097(3)
O(3)–Zn(1)–O(1)	106.96(13)	O(3)–Zn(1)–N(2)	127.27(14)
O(1)–Zn(1)–N(2)	121.87(13)	O(3)–Zn(1)–N(1)	106.47(12)
O(1)–Zn(1)–N(1)	104.63(12)	N(2)–Zn(1)–N(1)	80.72(14)
O(4)–Zn(2)–O(2)	108.74(13)	O(4)–Zn(2)–N(4)	122.54(14)
O(2)–Zn(2)–N(4)	125.08(14)	O(4)–Zn(2)–N(5)	102.88(12)
O(2)–Zn(2)–N(5)	106.97(12)	N(4)–Zn(2)–N(5)	80.93(14)
2			
Hg(1)–O(3)	2.079(7)	Hg(1)–N(2)	2.073(9)
Hg(1)–N(1)	2.523(9)	Hg(2)–O(6)	2.058(7)
Hg(2)–N(4)	2.081(9)	Hg(2)–N(5)	2.505(9)
O(3)–Hg(1)–N(2)	178.7(3)	O(3)–Hg(1)–N(1)	105.5(3)
N(2)–Hg(1)–N(1)	74.0(3)	O(6)–Hg(2)–N(4)	171.1(3)
O(6)–Hg(2)–N(5)	111.5(3)	N(4)–Hg(2)–N(5)	73.1(3)

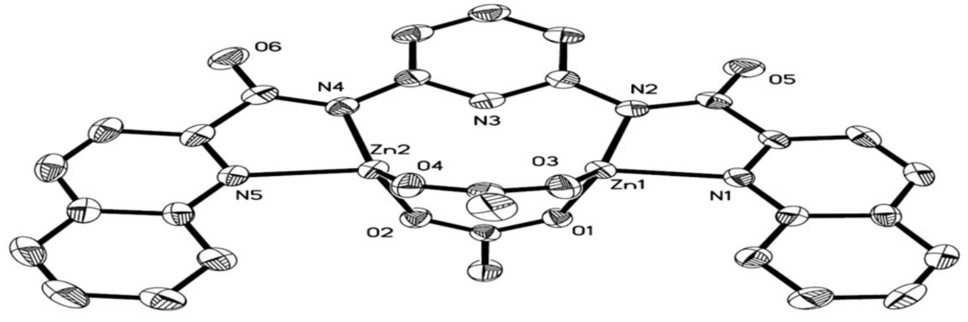

Figure 1. View of the dinuclear structure of **1** (hydrogens omitted for clarity).

3350 cm^{-1} is assigned to ν(N–H), characteristic of a strong hydrogen-bonding amide. However, in IR spectra of **1** and **2**, ν(N–H) bands around 3350 cm^{-1} vanish, indicating that the carboxamido groups were deprotonated during coordination. Therefore, each deprotonated ligand is L^{2-}. The strong absorptions at 1597 and 1344 cm^{-1} for **2** and the slightly weaker absorptions at 1598 and 1348 cm^{-1} for **1** are assigned to $\nu_{as}(COO^-)$ and $\nu_s(COO^-)$ of Ac, respectively.

3.2. *Structural descriptions of 1 and 2*

Complex **1** crystallizes in a monoclinic $C2/c$ space group. The structure of **1** is shown in figures 1–3. The asymmetric unit of **1** is built from two Zn(II) ions, one ligand L^{2-}, two Ac ions, one-and-a-half water molecule, and one-half methanol. As shown in figure 1, one doubly deprotonated L^{2-} in a bi-chelating μ-$kN,N':kN'',N'''$ bridges two Zn(II) centers to form the dinuclear structure, which is also doubly bridged by the two Ac ions

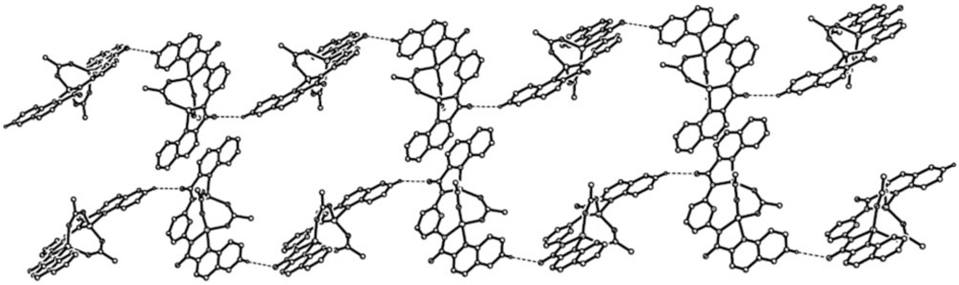

Figure 2. Through weak C–H $\cdots$ O hydrogen-bonding interactions, dinuclear units in **1** are linked into 1-D chains, which further stack by $\pi \cdots \pi$ stacking interactions between adjacent quinoline groups of L^{2-}.

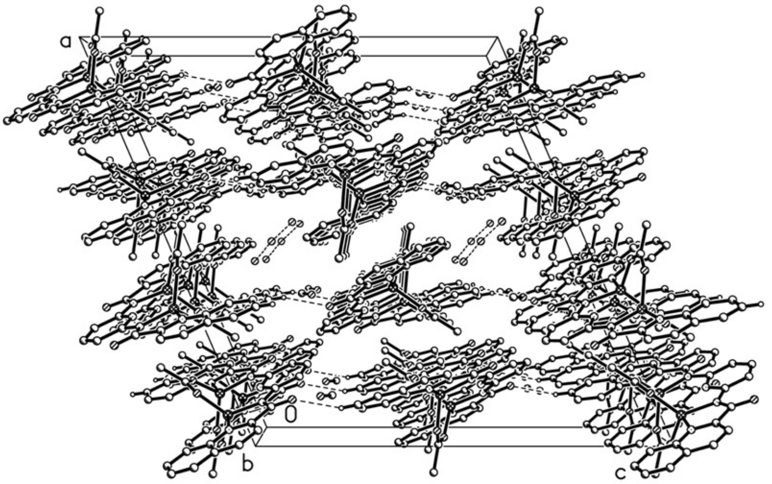

Figure 3. The stacking of **1**, showing free methanol molecules and trimeric water clusters residing in the crystal lattice.

in $\mu\text{-}k\mathrm{O}:k\mathrm{O}'$ coordination. Thus, every Zn(II) is a distorted tetrahedral coordination geometry ligated by two nitrogens from one quinoline-2-carboxamido of L^{2-} and two oxygens from two Ac ions. The sum of the bond angles of O(3)–Zn(1)–N(2) [127.27(14)°], O(3)–Zn(1)–O(1) [106.96(13)°], and N(2)–Zn(1)–O(1) [121.87(13)°] is of 356.10(40)° [close to 360°], indicating that Zn(1) is almost located at the plane O(1)O(3)N(2) [Zn(1) deviation from the basic plane O(1)O(3)N(2)Zn(1) is 0.165(0) Å]. For Zn(2), the sum of the bond angles of O(2)–Zn(2)–N(4) [125.08(14)°], O(2)–Zn(2)–O(4) [108.74(13)°], and N(4)–Zn(2)–O(4) [122.54(14)°] is 356.36(40)°, and Zn(2) deviation from the basic plane O(2)O(4)N(4)Zn(2) is −0.449(2) Å. In **1**, the Zn–O and Zn–N bond distances range from 1.935(3) to 2.102(3) Å and bond angles of N–Zn–O and O–Zn–O range from 102.88(12) to 127.27(14)°. Bond angles of N–Zn–N formed by the chelating coordination of quinoline-2-carboxamido of L to Zn(II) are 80.72(14)–80.93(14)°. As shown in scheme 1, free H_2L possesses C_{2v} symmetry, with a mirror plane m passing through nitrogen of the pyridine of H_2L to equally bisect

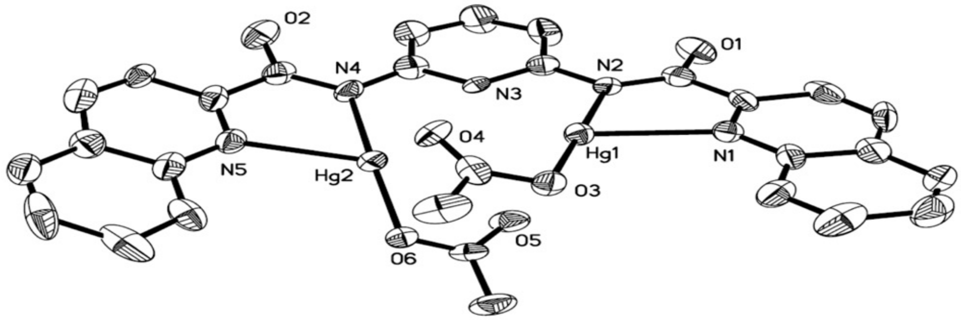

Figure 4. View of the basic dinuclear structure in **2** (hydrogens omitted for clarity).

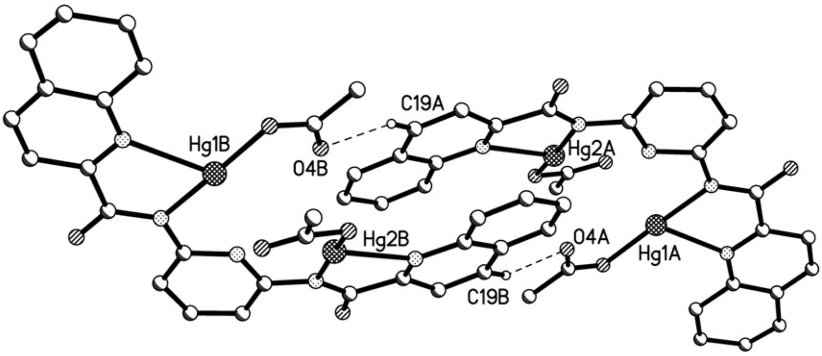

Figure 5. View of the hydrogen-bonded dimer in **2** (hydrogens omitted for clarity).

the molecule. Owing to coordination with zinc, the symmetry of deprotonated L^{2-} in **1** is lowered to pseudo C_2 symmetry, and the dihedral angle between the two quinoline-2-carboxamido groups is 6.5°. In the dinuclear unit, the separation of two Zn(II) ions by L^{2-} is 3.436 Å. Through weak $C-H\cdots O$ hydrogen-bonding interactions $[C-H\cdots O = 3.497\,\text{Å}]$, these dinuclear units are linked into 1-D chains which further pile by $\pi\cdots\pi$ stacking interactions between adjacent quinoline groups (figure 2). Free methanol and trimeric water clusters reside in the crystal lattice, as shown in figure 3.

Complex **2** crystallizes in a triclinic $P\bar{1}$ space group with the asymmetric unit consisting of two mercury(II) ions, one L^{2-}, two Ac ions, one methanol, and five water molecules. Similar to that in **1**, one doubly deprotonated L^{2-} bridges two Hg(II) centers to fabricate the dinuclear structure. In **2**, two Ac ions are terminal ligands to different Hg(II) centers, giving the rare distorted triangular or T-shaped coordination geometry (figure 4). The Hg–O bond distances range from 2.058(7) to 2.079(7) Å, whereas the Hg–N bond distances are very different. The two Hg–N bonds from carboxamido groups of L^{2-} ligating to Hg(1) and Hg(2) are 2.073(9) and 2.081(9) Å, respectively. However, the other two Hg–N bonds which form through nitrogen from quinoline of L^{2-} are obviously longer (Hg(1)–N(1) and Hg(2)–N(5) being 2.523(9) and 2.505(9) Å, respectively). In **2**, the intra-ring angles N(2)–Hg(1)–N(1) and N(4)–Hg(2)–N(5) are

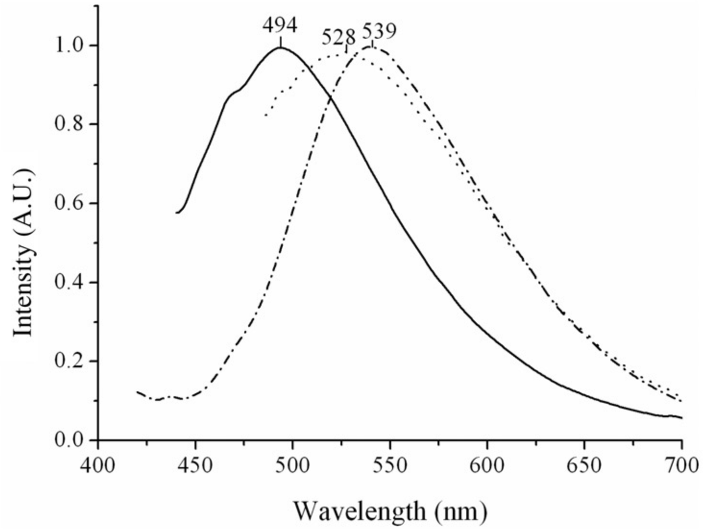

Figure 6. Solid-state photoluminescence spectra of H_2L, **1**, and **2** at room temperature (solid, dashed, and dotted lines for H_2L, **1**, and **2**, respectively).

74.0(3) and 73.1(3)°, respectively. The outer-ring angles N(2)–Hg(1)–O(3), O(3)–Hg(1)–N(1), O(6)–Hg(2)–N(4), and O(6)–Hg(2)–N(5) are 178.7(3), 105.5(3), 171.1(3), and 111.5(3)°, respectively. In **2**, the separation of two Hg(II) by L^{2-} is 3.678 Å, and the symmetry of deprotonated L^{2-} is C_1, with a bigger dihedral angle between the two quinoline-2-carboxamido groups of L^{2-} (21.6°). As shown in figure 5, two dinuclear units of **2** are paired by weak C–H ··· O hydrogen bonds to form a centrosymmetric tetra-metal complex [C–H ··· O = 3.332 Å]. The hydrogen-bonded tetra-metal units have $\pi \cdots \pi$ stacking interactions between adjacent quinoline groups of L^{2-} along the *a*-axis, with solvent molecules, such as methanol and trimeric water clusters between layers.

3.3. *Fluorescent properties*

Luminescence of metal–organic complexes is of great interest owing to their higher thermal stability and coordination-tunable emission wavelength and intensity compared with the emissions of the organic components [10, 19–21]. The solid-state photoluminescent behaviors of H_2L and its complexes **1** and **2** are investigated at room temperature. The emission spectra of **1** and **2** and free ligand H_2L are illustrated in figure 6. Free ligand H_2L is strongly photoluminescent and exhibits an intense cyan emission with a single broad band at $\lambda_{max} = 494$ nm, corresponding to excitation at $\lambda_{max} = 424$ nm. When excited at 419 nm, **1** and **2** display green emissions with emission maxima at 539 and 528 nm, respectively. In contrast to that of the free ligand H_2L, the emission wavelengths of **1** and **2** are red-shifted by 45 and 35 nm, respectively. The spectrum shapes of **1** and **2** resemble the free ligand H_2L. Probably, the emission of **1** and **2** is mainly due to a ligand-centered emission including significant charge transfer character induced by the polar cation as reported for Cd^{II} or other d^{10} metal complexes with N-donor ligands based on the position and shape of the emission bands [22–26].

From their structure analyses, the ligand-centered emission wavelengths of **1** and **2** are relative to the conformation of L^{2-} because the dihedral angle between the two quinoline-2-carboxamido groups of L^{2-} directly affect the extent of π conjugation of the system which mainly controls the energy gap between π^* and π molecular orbits of H_2L [27].

3.4. *Thermogravimetric analysis of 1*

The thermogravimetric (TG) curve of **1** was scanned from 30°C to 700°C on a STA 409 PC thermal analyzer under air with a flow rate of $30\,\mathrm{mL\,min^{-1}}$ and a heating rate of $10^\circ\mathrm{C\,min^{-1}}$ (Supplementary material). Weight loss starts at 47°C and to 382°C, weight loss is about 6.03%, corresponding to loss of free methanol and trimeric water clusters (Calcd 6.06%). Within the decomposition course, there is a clear step at about 200°C, and thus this decomposition can be divided into two steps. The first step occurs from 47°C to 180°C with a weight loss of 2.63%, indicating loss of free methanol (Calcd 2.26%), while the second step from 180°C to 382°C with a weight loss of 3.40% indicates loss of trimeric water clusters (Calcd 3.81%). Mass loss of about 69.43% from 382°C to 570°C may be caused by decomposition of the complex. The remaining 24.54% may be ZnO and unburned carbon (Calcd 22.54% for ZnO). Clearly, the basic dinuclear structure of **1** is very stable.

4. Conclusion

Through self-assembly of angular polytopic dipyridyl 2,6-*bis*(quinoline-2-carboxamido)pyridine (H_2L) with $ZnAc_2$ and $HgAc_2$, two new complexes **1** and **2** were obtained. X-ray single crystal structure analyses show that H_2L are doubly deprotonated and L^{2-} in $\mu\text{-}kN,N':kN'',N'''$ bridge two metal centers to form the dinuclear structures. Free ligand H_2L, **1**, and **2** emit bluish-green fluorescence with strong intensities. TG analysis of **1** shows that the dinuclear structure of **1** is stable at 382°C.

Supplementary material

Crystallographic information of **1** and **2** has been deposited with the Cambridge Crystallographic Data Centre, CCDC nos 753378 and 753379. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

Acknowledgments

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