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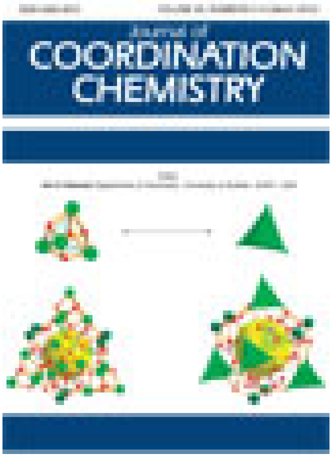

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Hydrothermal syntheses and structures of transition metal 2-(4,5-diphenyl-1H-imidazol-2-yl)benzoic acid complexes

Hui-Liang Wen [a], Tao-Tao Wang [a], Chong-Bo Liu [b], Min He [a] & Yuan-Xing Wang [a]

[a] State Key Laboratory of Food Science and Technology, Nanchang University, Nanchang 330047, People's Republic of China

[b] School of Environment and Chemical Engineering, Nanchang Hangkong University, Nanchang 330063, People's Republic of China

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Hydrothermal syntheses and structures of transition metal 2-(4,5-diphenyl-1*H*-imidazol-2-yl)benzoic acid complexes

HUI-LIANG WEN*†, TAO-TAO WANG†, CHONG-BO LIU‡, MIN HE† and YUAN-XING WANG†

†State Key Laboratory of Food Science and Technology, Nanchang University, Nanchang 330047, People's Republic of China
‡School of Environment and Chemical Engineering, Nanchang Hangkong University, Nanchang 330063, People's Republic of China

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Two new transition metal complexes, $[Zn(Hdiba)_2(H_2O)]\cdot H_2O$ (**1**) and $[Cu(Hdiba)_2]$ (**2**) ($H_2diba = $ 2-(4,5-diphenyl-1H-imidazol-2-yl)benzoic acid), were synthesized and characterized by IR, elemental analysis, and single-crystal X-ray diffraction. Complex **1** exhibits a monomeric structure, while **2** displays a dimeric structure. Both structures extend to 2-D supramolecular networks *via* hydrogen bonds. Thermal stabilities of **1** and **2** and photophysical properties of **1** are also discussed.

Keywords: 2-(4,5-Diphenyl-1*H*-imidazol-2-yl)benzoic acid; Transition metal complexes; Supramolecular network; Hydrothermal synthesis

1. Introduction

Architectures of supramolecular networks have aroused a great deal of interest for structural diversities and potential applications in magnetism, absorption, luminescence, and biological activities [1–6]. In particular, much attention has been focused on designing and synthesizing supramolecular coordination polymers based on multifunctional organic ligands with N- and O-donors [7–10]. Such compounds have multiple O- and N-coordination sites together with hydrogen-bond acceptors as well as hydrogen-bond donors, which are good candidates for assembly of high-dimensional 3d, 4f, or 3d–4f supramolecular networks. Compounds with imidazole ring system have received much attention due to rich pharmacological properties [11]. Many multi-substituted imidazoles are fungicides, herbicides, plant growth regulators, and therapeutic agents [12–14]. Derivatives of 2,4,5-triphenylimidazole (TPI) are widely used as fluorescent whiteners on textile [15], photographic materials [16], electroluminescent materials [17], and optical materials [18], largely owing to a large conjugated structure [19]. There are some reports on structures of TPI and derivatives [20, 21], with complexes with hydroxy-substituted TPI being characterized [22–25].

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*Corresponding author. Email: hlwen70@163.com

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There are no reports on the introduction of a carboxylate group to TPI. The carboxylate group possesses two O-coordination sites, which can increase the coordination ability of TPI with metal ions and also offers more hydrogen-bonding sites. In this article, we report the syntheses and crystal structures of two new transition metal complexes with 2-(4,5-diphenyl-1*H*-imidazol-2-yl)benzoic acid (H_2diba).

2. Experimental

2.1. *Materials and physical measurements*

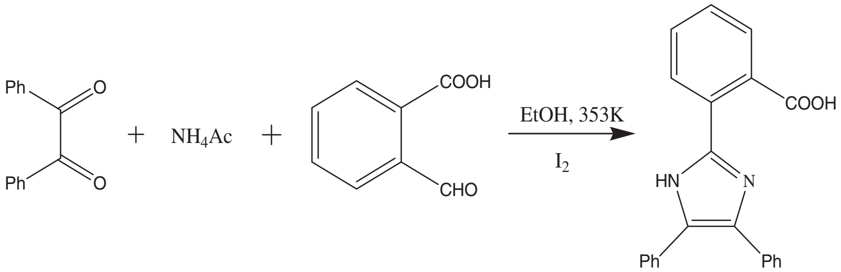
All materials and reagents except H_2diba were obtained commercially and used without purification. Elemental analyses of C, H, and N were carried out with a Flash EA 1112 Elementar analyzer. Infrared (IR) spectra were recorded with a Nicolet Avatar 360 FT-IR spectrometer using KBr pellets from 4000 to 400 cm^{-1}. ^{1}H NMR spectra were recorded on a Bruker Avance DPX 400 ultrashield instrument. Mass spectra were taken on an Agilent Liquid chromatography-mass spectrometry 6340 series instrument in the electrospray ionization (negative electrospray ionization) mode. Thermal stability studies were carried out on a Pyris diamond TGA/DTA thermal analyzer.

2.2. *Synthesis of 2-(4,5-diphenyl-1H-imidazol-2-yl)benzoic acid (H_2diba)*

H_2diba was synthesized by cyclization of diphenylethanedione with 2-carboxybenzaldehyde using HAc as solvent in a good yield [26] (scheme 1). IR data (KBr pellet, ν/cm^{-1}): 3392, 3060, 2924, 2854, 1560, 1509, 1458, 1436, 1395, 1372, 1319, 1295, 1248, 1176, 1135, 1111, 1077, 807, 768, 738, 706; ^{1}H NMR (DMSO-d_6) δ: 7.26(m, 2H, ArH), 7.39(m, 4H, ArH), 7.50(s, 5H, ArH), 7.61(s, 1H, ArH), 7.85(m, 1H, ArH), 8.27(m, 1H, ArH), 12.06(m, 1H, COOH), 13.2(m, 1H, N–H); MS (ESI) *m*/*z*: 339.19 ($M^+ - 1$).

2.3. *Syntheses of 1 and 2*

2.3.1. $[Zn(Hdiba)_2(H_2O)]\cdot H_2O$ (1). A mixture of 0.1 mmol $ZnSO_4 \cdot 7H_2O$, 0.1 mmol H_2diba, and 0.625 mL 0.65 mol L^{-1} NaOH aqueous solution in 10 mL water was sealed in a 25 mL Teflon-lined stainless reactor and heated at 160°C for 72 h under autogenous



Scheme 1. Synthesis route of H_2diba.

pressure then cooled at 10°C h^{-1} to 80°C followed by slow cooling to room temperature. Upon cooling to room temperature, the desired product appeared as colorless block crystals in *ca* 52% yield. Anal. Calcd for $C_{44}H_{36}ZnN_4O_6$ (780.12) (%): C, 67.74; N, 7.18; H, 4.65. Found (%): C, 67.49; N, 7.02; H, 4.98. IR data (KBr pellet, ν/cm^{-1}): 3418 s, 3061 w, 2924 w, 1579 vs, 1421 m, 1386 m, 764 w, 694 m.

2.3.2. [Cu(Hdiba)$_2$] (2). A mixture of 0.1 mmol $CuCl_2$, 0.1 mmol H_2diba, and 0.625 mL 0.65 mol L^{-1} NaOH aqueous solution in 6 mL water and 4 mL isopropanol was sealed in a 25 mL Teflon-lined stainless reactor and heated at 120°C for 72 h under autogenous pressure then cooled at 10°C h^{-1} to 80°C followed by slow cooling to room temperature. Upon cooling to room temperature, the desired product appeared as dark purple rhombus crystals in *ca* 47% yield. Anal. Calcd for $C_{44}H_{30}CuN_4O_4$ (742.26) (%): C, 71.19; H, 4.07; N, 7.55. Found (%): C, 71.22; H, 4.31; N, 7.13. IR data (KBr pellet, ν/cm^{-1}): 1578 vs, 1423 m, 1384 m, 767 w, 691 m.

2.3.3. X-ray crystallography. Single-crystal X-ray data of **1** and **2** were collected using a Bruker APEX II area detector diffractometer with graphite-monochromated Mo-Kα radiation ($\lambda = 0.71073$ Å). Semi-empirical absorption corrections were applied to **1** and **2** using SADABS. The structures were solved by direct methods using SHELXS-97 [27] and refined by full-matrix least-squares on F^2 using SHELXS-97 [28]; all non-hydrogen atoms were refined anisotropically. Hydrogen atoms attached to C and N were placed in geometrically calculated positions; the water hydrogen atoms were located in difference Fourier maps and refined with O–H distance restraints of 0.82–0.83 Å.

3. Results and discussion

3.1. *Structural descriptions*

Crystal data of **1** and **2** are listed in table 1 and selected bond lengths in table 2. Hydrogen-bond data are listed in table 3. In **1** and **2**, H_2diba ligands only adopt one kind of coordination (scheme 2).

3.2. *The structure of [Zn(Hdiba)$_2$(H$_2$O)] • H$_2$O (1)*

The structure of **1** is composed of a repeating unit of $[Zn(Hdiba)_2(H_2O)] \cdot H_2O$. Each Zn(II) is in an O_3N_2 environment provided by two $Hdiba^-$ ligands with two carboxylate oxygen atoms and two imidazole nitrogen atoms and one water molecule in a distorted trigonal bipyramid geometry with O1, N1, and N3 comprising the equatorial plane, O4 and O5 occupying the axial positions (figure 1a). The H_2diba ligands adopt an N,O-bidentate chelating coordination mode (scheme 2). In **1**, there are two orientations for H_2diba with dihedral angle between the planes of imidazole rings of *ca* 44.9°. In **1**, each monomeric entity is linked with neighbors *via* O2–H4W···O3 and N2–H2D···O3 hydrogen bonds into chains (figure 1b), which are further joined to a 2-D supramolecular network through N4–H4D···O3 and N4–H4D···O4 hydrogen bonds along the *c*-axis (figure 1c).

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

	1	**2**
Empirical formula	$C_{44}H_{34}N_4O_6Zn$	$C_{44}H_{30}N_4O_4Cu$
Formula weight	780.12	742.26
Temperature (K)	293(2)	293(2)
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Unit cell dimensions (Å, °)		
a	8.066(8)	11.946(3)
b	8.636(9)	13.040(3)
c	27.304(3)	18.181(5)
α	90.593(10)	101.317(4)
β	93.584(10)	99.015(4)
γ	101.903(10)	100.612(4)
Volume (Å^3), Z	1856.8(3), 2	2674.5(12), 3
Calculated density (Mg m^{-3})	1.395	1.383
Absorption coefficient (mm^{-1})	0.717	0.663
F(000)	808	1149
θ range for data collection (°)	2.24–25.50	2.31–25.50
Reflections/collected	12,553/6703	20,520/9885
Goodness-of-fit on F^2	1.054	0.996
Unique [R_{int}]	0.0230	0.0722
R_1, wR_2 [$I > 2\sigma(I)$]	0.0377, 0.0827	0.0730, 0.1836
R_1, wR_2 (all data)	0.0506, 0.0885	0.1232, 0.2221

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

1			
Zn(1)–O(1)	2.035(17)	Zn(1)–N(1)	2.037(19)
Zn(1)–O(4)	2.150(19)	Zn(1)–N(3)	2.049(2)
Zn(1)–O(5)	2.057(17)		
O(1)–Zn(1)–O(4)	75.96(7)	N(1)–Zn(1)–O(4)	90.08(8)
O(1)–Zn(1)–O(5)	89.84(7)	N(3)–Zn(1)–O(4)	95.11(8)
O(1)–Zn(1)–N(1)	119.58(8)	N(1)–Zn(1)–O(5)	104.47(7)
O(1)–Zn(1)–N(3)	126.74(8)	N(3)–Zn(1)–O(5)	86.57(8)
O(5)–Zn(1)–O(4)	163.47(7)	N(1)–Zn(1)–N(3)	112.70(8)
2			
Cu(1)–O(1)	1.914(3)	Cu(2)–O(5)	1.888(4)
Cu(1)–O(3)	1.923(4)	Cu(2)–O(5)#1	1.888(4)
Cu(1)–N(1)	1.966(4)	Cu(2)–N(5)	1.990(4)
Cu(1)–N(3)	1.972(4)	Cu(2)–N(5)#1	1.990(4)
O(1)–Cu(1)–O(3)	93.29(17)	O(5)–Cu(2)–O(5)#1	179.998(1)
O(1)–Cu(1)–N(1)	88.50(16)	O(5)–Cu(2)–N(5)	88.44(16)
O(1)–Cu(1)–N(3)	155.50(17)	O(5)–Cu(2)–N(5)#1	91.56(16)
O(3)–Cu(1)–N(1)	155.34(17)	O(5)#1–Cu(2)–N(5)	91.56(16)
O(3)–Cu(1)–N(3)	89.33(16)	O(5)#1–Cu(2)–N(5)#1	88.44(16)
N(1)–Cu(1)–N(3)	99.14(17)	N(5)–Cu(2)–N(5)#1	179.998(1)

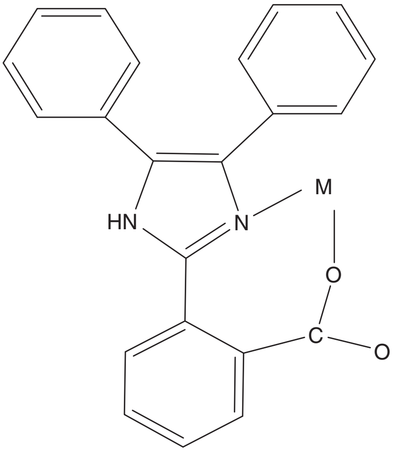
Symmetry code: #1: $-x + 1$, $-y$, $-z + 1$.

3.3. *The structure of [Cu(Hdiba)$_2$] (2)*

The structure contains two crystallographically independent Cu(II) ions at 10.03 Å (figure 2a). Cu(II) in **2** are four-coordinated, rare in coordination chemistry [29–31]. Cu1 and Cu2 are coordinated to two carboxylate oxygen atoms and two imidazole nitrogen atoms from two H_2diba ligands with small differences between Cu(1)–O and Cu(2)–O, Cu(1)–N and Cu(2)–N bond lengths. In **2**, Cu2, which is located on an

Table 3. Hydrogen-bonding geometry (Å and °) for **1** and **2**.

D–H	d(D–H)	d(H···A)	∠DHA	d(D···A)	A	Symmetry code
1						
O(1)–H(1W)	0.82	1.78	169.0	2.594(3)	O(2)	
O(2)–H(3W)	0.83	2.11	147.0	2.836(3)	O(5)	
O(2)–H(4W)	0.83	2.03	162.0	2.831(3)	O(3)	$x, y-1, z$
N(2)–H(2D)	0.86	1.82	173.0	2.676(2)	O(6)	$x, y+1, z$
N(4)–H(4D)	0.86	2.05	162.0	2.881(3)	O(3)	$x+1, y, z$
N(4)–H(4D)	0.86	2.56	142.0	3.281(3)	O(4)	$x+1, y, z$
2						
N(2)–H(2)	0.86	1.83	176.0	2.685(6)	O(6)	
N(4)–H(4D)	0.86	1.90	160.0	2.719(5)	O(4)	$1-x, 2-y, 2-z$
N(6)–H(6)	0.86	1.98	174.0	2.840(6)	O(2)	$1-x, 1-y, 1-z$



Scheme 2. Coordination mode of H_2diba.

inversion center, exhibits a quadrilateral configuration while Cu1 displays a distorted tetrahedral configuration. H_2diba ligands adopt one coordination mode, as for **1**. In **2**, the H_2diba have three orientations with dihedral angles between the planes of imidazole rings of *ca* 49.2°, 66.2°, and 34.8°.

Intramolecular N2–H2 ··· O6 hydrogen bonds join [Cu1N2O2] and [Cu2N2O2] units to form dimers, which are further linked to a chain structure *via* double N4–H4D ··· O4 hydrogen bonds (figure 2b). Adjacent chains are joined to form a 2-D network *via* N6–H6 ··· O2 (symmetry code: $-x+1, -y+1, -z+1$) hydrogen bonds (figure 2c).

Whereas the ionic radii of Cu(II) and Zn(II) are nearly the same, the coordination numbers of Cu(II) and Zn(II) are four and five, respectively, with the mean Cu–O and Cu–N bond distances of 1.903 and 1.980 Å being shorter than the average Zn–O and Zn–N bond distances of 2.080 and 2.043 Å, respectively. In **1** and **2**, each H_2diba adopts an N,O-chelating coordination to one metal ion and forming monomeric **1** and dimeric **2**. Both solids result in 2-D hydrogen-bonded supramolecular frameworks.

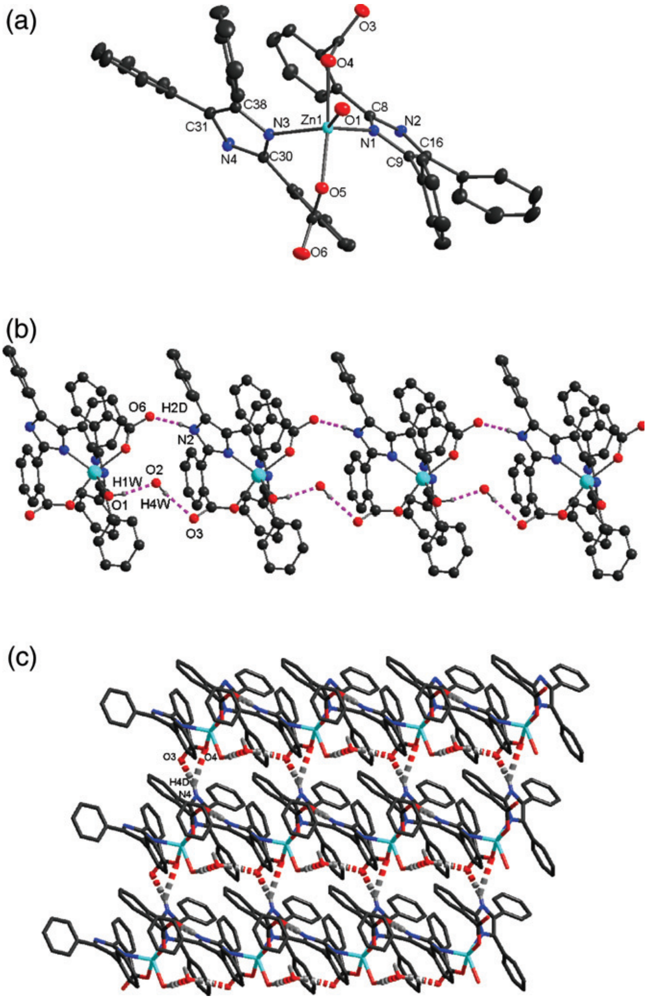


Figure 1. (a) The coordination environment of Zn^{2+} in **1** with thermal ellipsoids at 20% probability; (b) view of 1-D chain structure of **1** along the *c*-axis; and (c) view of the 2-D supramolecular structure of **1** along the *c*-axis. All hydrogen atoms not involved in hydrogen-bonding are omitted for clarity, hydrogen-bonding interactions are indicated as dotted lines.

3.4. *Thermogravimetric analysis of 1 and 2*

The thermogravimetric (TG) curve of **1** exhibits two weight losses from 40°C to 800°C. The first weight loss of 4.16% from 40°C to 133°C corresponds to loss of two water molecules per asymmetric unit (Calcd: 4.61%). The compound is stable to 280°C, with the second weight loss of 88.82% (Calcd: 89.57%) from 280°C to 800°C corresponding to decomposition of the organic groups and the final residual being ZnO. Complex **2** exhibits a weight loss from 250°C with the final pyrolysis being completed at 790°C. The loss has weight change of 88.9%; CuO (Calcd 89.3%). The thermal stability of **2** is, thus, much higher than that of **1** with uncoordinated and coordinated water molecules.

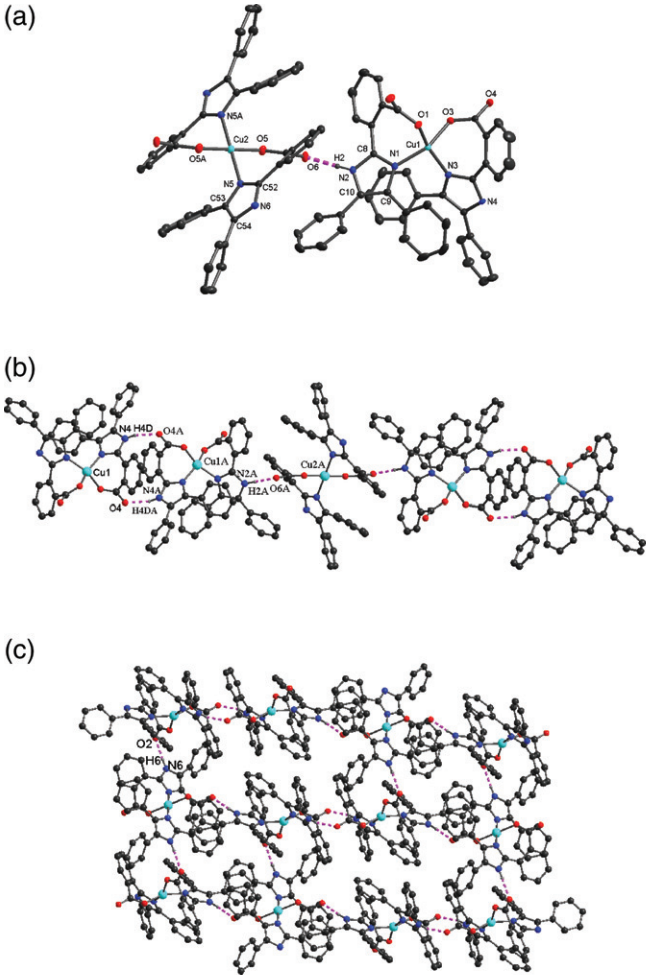


Figure 2. (a) The coordination environment of Cu^{2+} in **2** with thermal ellipsoids at 20% probability; (b) view of 1-D chain structure of **2** along the *c*-axis; and (c) view of the 2-D supramolecular structure of **2** along the *c*-axis. All hydrogen atoms not involved in hydrogen-bonding are omitted for clarity, hydrogen-bonding interactions are indicated as dotted lines.

3.5. *Luminescence of 1*

Complex **1** exhibits solid-state luminescence, with the emission spectra of **1** and H_2diba in the solid state being investigated (figure 3). Complex **1** exhibits an emission maximum at 390 nm upon excitation at 274 nm with a blue shift of 39 nm in contrast to H_2diba. The resemblance of the emission spectrum of **1** and that of free H_2diba indicates that the luminescence of **1** is due to a $\pi^* \rightarrow \pi$ electronic transition of H_2diba [32, 33]. The fluorescence intensities of **1** are *ca* two times larger than those of free H_2diba. Enhancement and significant blue shift in luminescence of **1** compared to free H_2diba may, therefore, be attributed to H_2diba chelating to Zn^{2+}.

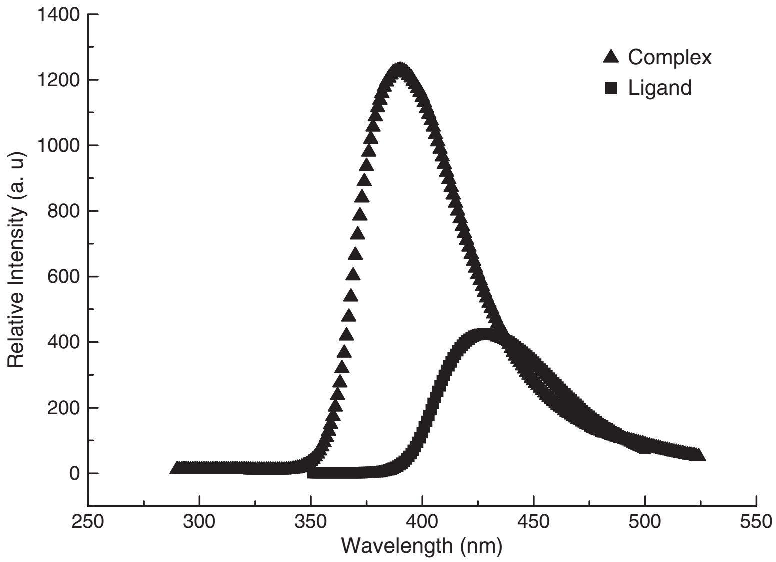


Figure 3. Emission spectrum of **1** and H_2diba.

4. Conclusion

Two new transition metal supramolecular complexes with a new imidazole carboxylate have been obtained. The single-crystal X-ray diffraction results indicate that different metal (II) ion, counter anions and solvent take important roles in the formation of diverse structures of **1** and **2**. TG analyses of **1** and **2** show water molecules affect the thermal stabilities of **1**.

Supplementary material

Crystallographic data for **1** and **2** have been deposited with the Cambridge Crystallographic Data Center with deposition numbers CCDC 842687 and 842688, respectively.

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