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Syntheses, crystal structures and luminescent properties of two new 1D d^{10} coordination polymers constructed from 2,2′-bibenzimidazole and 1,4-benzenedicarboxylate

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Abstract

Two novel interesting d^{10} metal coordination polymers, $[Zn(H_2bibzim)(BDC)]_n$ (**1**) and $[Cd(H_2bibzim)(BDC)]_n$ (**2**) [$H_2bibzim$ = 2,2′-bibenzimidazole, BDC = 1,4-benzenedicarboxylate] have been synthesized under solvothermal conditions and structurally characterized. Both **1** and **2** are constructed from infinite neutral zigzag-like one-dimensional (1D) chains. The π–π interactions and interchain hydrogen-bonding interactions further extend the 1D arrangement to generate a 3D supramolecular architecture for **1** and **2**. Both complexes have high thermal stability and display strong blue fluorescent emissions in the solid state upon photo-excitation at 365 nm at room temperature. They are the first two examples that 2,2′-bibenzimidazole has been introduced into the d^{10} coordination polymeric framework.

Keywords: 2,2′-Bibenzimidazole; X-ray structure; Zn(II) complex; Cd(II) complex

1. Introduction

Recently, the field of crystal engineering and rational design of novel inorganic–organic hybrid materials have attracted great interest from chemists [1,2] and considerable efforts have been focused on the design, synthesis and characterization of novel multidimensional structures [3,4], not only because of their intriguing variety of architectures and topologies, but also because of their versatile potential applications as ion exchange, catalysis and for the development of optical, electronic and magnetic devices [5,6]. The ligand 2,2′-bibenzimidazole($H_2bibzim$) contains multiple nitrogen-donor sites with the possibility of reversible protonation and deprotonation properties [7] and can be coordinated to a transition metal in non-deprotonated (neutral, $H_2bibzim$), mono-deprotonated (monoanion, $Hbibzim^-$) and di-deprotonated (dianion, $bibzim^{2-}$) forms, which has been utilized in supramolecular chemistry for the preparation of multidimensional hydrogen-bonded networks [8]. In the formally neutral form, two nitrogen donors in coplanar rings can adopt a *syn* orientation, which is similar to the chelate mode of 2,2′-bipyridine. However, $H_2bibzim$ differs from bipyridine by the presence of five-membered rings, which change the bite angle for chelate, resulting in the formation of a rich variety of oligomeric or polymeric structures [9,10]. In addition, the residual N–H entities form particular hydrogen-bond interactions from which supramolecular assemblies can be constructed. As a rigid and versatile bridging ligand, 1,4-benzenedicarboxylate (BDC) has been extensively studied for designing new inorganic–organic hybrid materials, which is also a good candidate bearing a complementary functional group linked to N–H entities [11,12]. Therefore, with the aim of preparing novel materials with beautiful architecture and good physical properties, we start to synthesize new coordination polymers constructed from 2,2′-bibenzimidazole and 1,4-BDC (Scheme 1). In the present paper, we report the syntheses, structural characterizations, and photoluminescence properties of two new one-dimensional coordination polymer $[Zn(H_2bibzim)(BDC)]_n$ (**1**) and $[Cd(H_2bibzim)$

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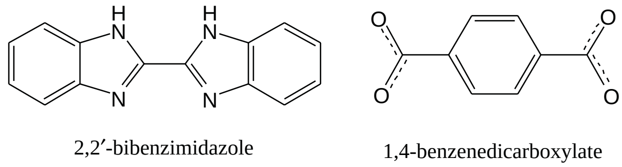
2,2′-bibenzimidazole 1,4-benzenedicarboxylate

Scheme 1.

(BDC)]$_n$ (**2**). To the best of our knowledge, they are the first two examples that 2,2′-bibenzimidazole has been introduced into the d^{10} coordination polymers.

2. Experimental

The reagents and solvents employed were commercially available and used as received without further purification. Ligand H_2bibzim was prepared according to the previously reported procedure [13]. The C, H, and N microanalyses were carried out with a Perkin-Elmer 240 elemental analyzer. The IR spectra were recorded on KBr discs on a Bruker Vector 22 spectrophotometer in the 4000–400 cm^{-1} region. Luminescence spectra for the solid samples were recorded with a Hitachi 850 fluorescence spectrophotometer. Thermogravimetric analysis was performed on a simultaneous SDT 2960 thermal analyzer. Powder samples of complexes **1** and **2** were heated from room temperature to 800 °C under flowing N_2 at a heating rate of 10 °C/min. Powder X-ray diffraction patterns were recorded on a RigakuD/max-RA rotating anode X-ray diffractometer with graphitemonochromatic Cu*K*α ($\lambda = 1.542$ Å) radiation at room temperature.

2.1. Preparation of [Zn(H_2bibzim)(BDC)]$_n$ 1

A mixture of $Zn(NO_3)_2 \cdot 6H_2O$ (0.5 mmol, 148.8 mg), H_2bibzim (0.5 mmol, 117.3 mg), H_2BDC (0.5 mmol, 82.4 mg), NaOH (1.0 mmol, 40.3 mg) and CH_3CN/H_2O (12 mL, $V/V = 1{:}2$) was placed in a parr Teflon-lined stainless steel vessel (25 cm^3), and then the vessel was sealed and heated at 120 °C for 3 days. After the mixture was slowly cooled to room temperature, yellow crystals of **1** were obtained (yield: 84% based on Zn). Anal. Calcd for $C_{22}H_{14}N_4O_4Zn$: C 56.98, H 3.04, N 12.08. Found: C 56.93, H 2.98, N 12.04%. IR spectrum (cm^{-1}): 3328 (m), 3074 (m), 1618 (m), 1560 (s), 1507 (m), 1407 (s), 1395 (s), 1353 (m), 1298 (m), 1148 (m), 1039 (m), 1012 (m), 896 (m), 847 (s), 765 (s), 738 (s), 621 (m), 584 (m), 532 (m), 427(w).

2.2. Preparation of [Cd(H_2bibzim)(BDC)]$_n$ 2

The synthesis was similar to that described for **1** except using $CdCl_2 \cdot 2H_2O$ (0.5 mmol) instead of $Zn(NO_3)_2 \cdot 6H_2O$, and yellow crystals of **2** were obtained (yield: 54% based on Cd). Anal. Calcd for $C_{22}H_{14}CdN_4O_4$: C 51.73, H 2.76, N 10.97. Found: C 51.71, H 2.79, N 10.93%. IR spectrum (cm^{-1}): 3448 (m), 3134 (m), 3103 (m), 1618 (m), 1577 (m), 1560 (s), 1545 (s), 1405 (s), 1395 (s), 1376 (s), 1299 (w), 1148 (m), 1034 (m), 1014 (m), 898 (m), 843 (s), 765 (m), 735 (s), 621 (w), 579 (w), 531 (m), 423 (w).

2.3. X-ray crystal structure determinations

Intensities of complexes **1** and **2** were collected on a Siemens SMART-CCD diffractometer with graphite-monochromatic Mo*K*α radiation ($\lambda = 0.71073$ Å) using the SMART and SAINT programs [14]. The structures were solved by direct methods and refined on F^2 using full-matrix least-squares methods with SHELXTL version 5.1 [15]. Anisotropic thermal parameters were refined for the non-hydrogen atoms. Hydrogen atoms attached to carbon and nitrogen were positioned geometrically (C–H = 0.93 Å, N–H = 0.86 Å), and included in the refinement in a riding model approximation with an isotropic thermal displacement parameter fixed at 1.2 times U_{eq} of the atom to which they are attached. Crystallographic data and other pertinent information for **1** and **2** are summarized in Table 1. Selected bond lengths and bond angles with their estimated standard deviations are listed in Table 2.

3. Results and discussion

3.1. Crystal structures of [Zn(H_2bibzim)(BDC)]$_n$ (1) and [Cd(H_2bibzim)(BDC)]$_n$ (2)

The crystal structure of **1** is constructed from neutral zigzag-like 1D chain consisting of [Zn(H_2bibzim)(BDC)] entities. Each Zn^{2+} ion adopts a distorted trigonal

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

Complex	1	2
Empirical formula	$C_{22}H_{14}N_4O_4Zn$	$C_{22}H_{14}CdN_4O_4$
Formula weight	463.74	510.77
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/c$
a (Å)	7.3216(9)	7.4326(14)
b (Å)	21.936(3)	22.175(4)
c (Å)	11.603(1)	11.752(2)
β (deg)	97.663(3)	93.961(4)
V(Å^3)	1846.9(4)	1932.3(6)
Z	4	4
Dc (g/cm^3)	1.668	1.756
μ (mm^{-1})	1.371	1.170
F(000)	944	1016
Crystal size (mm^3)	0.15 × 0.15 × 0.10	0.15 × 0.10 × 0.10
2*θ* max (deg)	54.00	52.00
Index ranges	$-9 \leqslant h \leqslant 9$	$-8 \leqslant h \leqslant 9$
	$-27 \leqslant k \leqslant 17$	$-27 \leqslant k \leqslant 27$
	$-14 \leqslant l \leqslant 14$	$-12 \leqslant l \leqslant 14$
Reflns collected	10637	10310
Indep reflns	4005	3784
GOF on F^2	1.10	1.12
R_1 [$I > 2\sigma(I)$]	0.0505	0.0468
wR_2	0.0995	0.0889
Residual electron density (e Å^{-3})	−0.61, 0.29	−0.67, 0.50

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

[Zn(H$_2$bibzim)(BDC)] (**1**)			
Zn1–O1	2.168(2)	Zn1–N4	2.086(2)
Zn1–O2	2.154(2)	Zn1–O3[a]	2.092(2)
Zn1–N1	2.106(2)	Zn1–O4[a]	2.235(2)
O1–Zn1–O2	60.87(8)	N1–Zn1–N4	79.68(9)
O3[a]–Zn1–O4[a]	60.69(8)		
[Cd(H$_2$bibzim)(BDC)] (**2**)			
Cd1–O1	2.306(3)	Cd1–N4	2.270(3)
Cd1–O2	2.353(3)	Cd1–O3[b]	2.329(4)
Cd1–N1	2.311(3)	Cd1–O4[b]	2.313(3)
O1–Cd1–O2	56.16(10)	O3[b]–Cd1–O4[b]	56.04(10)
N1–Cd1–N4	73.72(12)		

[a] Equivalent atoms generated by $1+x$,$1/2-y$,$1/2+z$.
[b] Equivalent atoms generated by $1+x$, $3/2-y$, $1/2+z$.

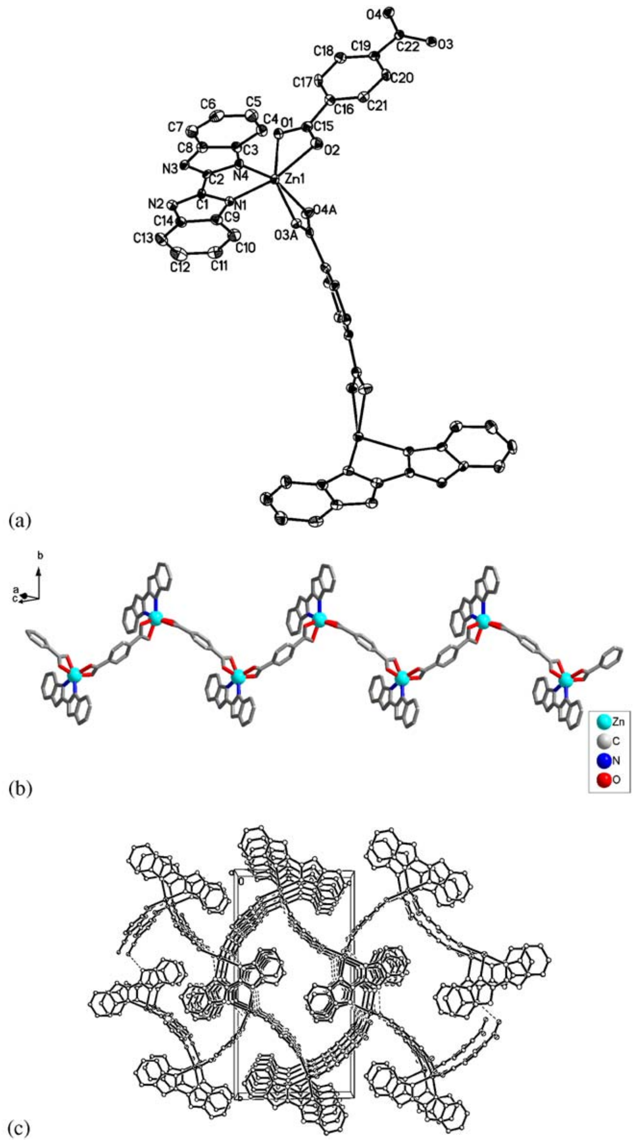

Fig. 1. (a) Coordination environment of Zn(II) metal ion in **1**. (b) A 1D zigzag conformation of **1** with hydrogen atoms omitted for clarity. (c) Crystal packing diagram for complex **1** on *bc* plane with hydrogen bonds indicated by dashed lines.

prismatic coordination geometry of {ZnN_2O_4}, defined by two nitrogen donors from one non-deprotonated bidentate H$_2$bibzim ligand, four oxygen donors from two different BDC anions, as indicated in Fig. 1a. The Zn–N bond lengths are 2.086(2) and 2.107(2) Å, which are comparable to those detected in [Zn(H$_2$biim)(H$_2$O)](OAc)$_2$ · OHCH$_2$CH$_2$OH [16] and slightly shorter than the apical distance (2.184(5)–2.265(5) Å) in [Zn$_2$(μ-H$_2$Biim)(H$_2$Biim)$_4$](ClO$_4$)$_4$ · 3H$_2$O [17]. Meanwhile, the Zn–O bond distances fall in the range from 2.092(2) to 2.235(2) Å. The N–Zn–N angle is 79.69(9)° whereas the O–Zn–O angles formed by chelating carboxylate range between 60.68(8)° and 150.88(8)° (Table 3).

Compound **2** is isostructural to compound **1**; each Cd^{2+} ion adopts similar distorted trigonal prismatic coordination geometry of {CdN_2O_4}. The Cd–N bond lengths are 2.270(3) and 2.311(3) Å, which are consistent with the reported distances for [CdBr$_2$(H$_2$biim)]$_n$ [18], with the N–Cd–N angle of 73.7(1)°. The Cd–O bond distances are in the range from 2.306(3) to 2.353(3) Å with the O–Cd–O angles varying from 56.04(10)° to 151.34(9)°.

The two carboxylate groups are out of the plane of correspondingly linking phenyl rings, with the dihedral angles between them being ca. 13° and 159° for **1** as well as being ca. 14 and 30° for **2**, respectively. These distortions are reflected upon the 1D-arrangment, which maintains a zigzag conformation along the growing axis, as depicted in Fig. 1b. Each BDC anion connects two zinc centers with the Zn···Zn separations of 10.524(1) Å. The Zn–Zn–Zn angles defined by the orientation of the two adjacent BDC anions in the [Zn(H$_2$bibzim)(BDC)] building units are 90°, which are far away from the ideal value of 120°. Similarly, for **2**, the Cd···Cd separations bridged by BDC anion are 10.905(1) Å. The Cd–Cd–Cd angles are 90°, which are identical to those observed in **1**.

For H$_2$bibzim ligand, ring closure induces in-plane distortions to improve the overlap between the metal orbitals and the ligand δ lone pairs, leading to a large difference between the N1–C1–C2 and N2–C1–C2 angles

Table 3
Distances (Å) and angles (deg) of hydrogen bonding for complexes **1** and **2**

D–H···A	*d*(D–H)	*d*(H···A)	*d*(D···A)	< (D–H···A)
[Zn(H$_2$bibzim)(BDC)] (**1**)				
N2–H2A···O1[a]	0.8600	2.0100	2.769(3)	147.00
N3–H3A···O3[b]	0.8600	2.1200	2.799(3)	136.00
C17–H17···O3[c]	0.9300	2.5300	3.328(4)	144.00
[Cd(H$_2$bibzim)(BDC)] (**2**)				
N2–H2A···O4[d]	0.8600	2.0500	2.768(4)	141.00
N3–H3A···O1[e]	0.8600	2.0500	2.788(4)	143.00
C18–H18–O1[f]	0.9300	2.5400	3.338(4)	144.00

[a] Equivalent atoms generated by $-x$, $1-y$, $2-z$.
[b] Equivalent atoms generated by $-x$, $1/2+y$, $3/2-z$.
[c] Equivalent atoms generated by x,$1/2-y$,$1/2+z$.
[d] Equivalent atoms generated by $-x$, $-1/2+y$, $1/2-z$.
[e] Equivalent atoms generated by $-x$, $1-y$, $1-z$.
[f] Equivalent atoms generated by x, $3/2-y$, $-1/2+z$.

(~117° versus ~129°) for **1**. Obviously, for **2**, the difference between the two angles (~119° versus ~127°) is somewhat smaller than that in **1**. The central C1–C2 bond length found for **1** is 1.468(3) Å and for **2** is 1.447(5) Å, which is slightly longer than those observed in free biimidazole [19] and $[Ni(H_2biim)_2(H_2O)_2](NO_3)_2$ [20] (1.423, and 1.441 Å, respectively). For both compounds, although the two independent benzimidazole rings remain individually planar, the H_2bibzim molecule is bent slightly about the C1–C2 bond, leading to a dihedral angle of 4.6° for **1** and 4.1° for **2** between these benzimidazole rings. The 1D systems run in an almost parallel alignment to each other, allowing face-to-face π–π interactions between adjacent benzene rings (3.748 Å for **1**, 3.722 Å for **2**) and imidazole rings (3.745 Å for **1**, 3.657 Å for **2**) to sustain the 2D-assembly extended in the *ab* plane, as illustrated in Fig. 2.

As anticipated, the couple of N–H groups of each bibenimidazole molecule are involved in hydrogen bonds with the carboxylate oxygen atoms. For **1**, the $N2\cdots O1^a$ distance of 2.769(3) Å and $N3\cdots O3^b$ distance of 2.799(3) Å correspond to strong interactions. The four atoms N(2), O(1), N(3), and O(3) are almost coplanar, with a mean deviation of 0.0122 Å. Furthermore, the O3 atom also has weak interaction with C17 atom via a nonclassical hydrogen bond, $C17–H17\cdots O3^c$ distance of 3.328(4) Å. As a consequence, the π–π interactions and interchain hydrogen-bonding interactions further extend the 1D arrangement to generate a 3D supramolecular architecture (Fig. 1c).

For **2**, the $N2\cdots O4^d$ distance is 2.768(4) Å and $N3\cdots O1^e$ distance is 2.788(4) Å with the $N–H\cdots O$ systems not deviated much from linearity (~143°), also indicating strong interactions. The four atoms N(2), O(4), N(3), and O(1) are not coplanar, with a mean deviation of 0.4795 Å, which is distinctly larger than those in **1**. In addition, there also exist interlayer weak hydrogen bond interactions with $C18–H18\cdots O1^f$ distance of 3.338(4) Å. Doubtless, the combination of π–π overlap and interchain hydrogen-bonding interactions further extend the 1D chain to afford a 3D framework, as displayed in Fig. S1.

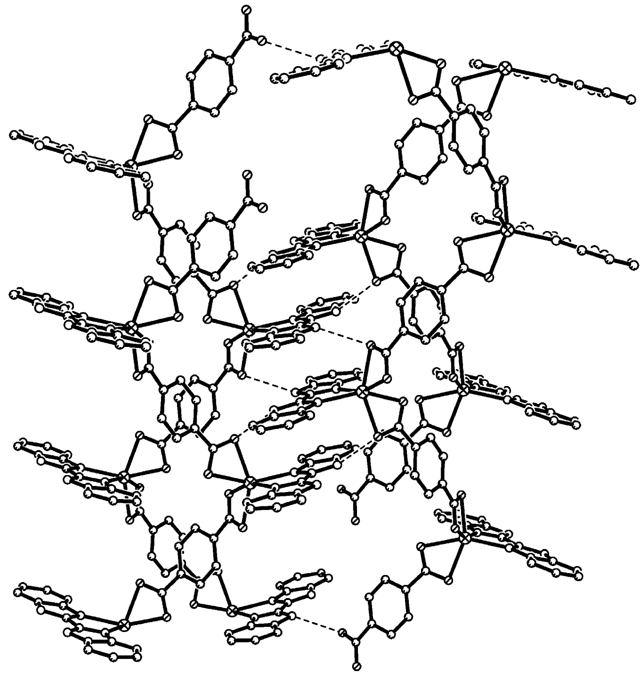

Fig. 2. Face-to-face π–π interactions between adjacent benzene rings and imidazole rings from two nearest 1D zigzag chains for **2**.

3.2. IR spectra

The broad absorption bands of 3328 and 3074 cm^{-1} for **1** and the 3448, 3134 and 3103 cm^{-1} for **2** in the IR spectra can be assigned to the ν(N–H) stretching vibrations [17,21]. The coordination fashion of the H_2BDC moiety to the metal atom in **1** and **2** is proved by the shifts of the stretching vibrations ν_{as} and ν_s of the carboxyl group. The difference between ν_{as}(OCO) (1560 cm^{-1}) and ν_s(OCO) (1407 cm^{-1}) is 153 cm^{-1} for **1**, which is indicative of a chelating carboxyl group. For **2**, the difference between ν_{as}(OCO) (1545 cm^{-1}) and ν_s(OCO) (1405 cm^{-1}) is 140 cm^{-1} also indicative of a chelating one [22], which are consistent with the crystal structures. For the two compounds, the difference in the increment extent of the ligand conformational rigidity and conjugation due to their coordination to different metal ions may exert a little influence on their stretching modes.

3.3. XRD spectra and thermogravimetric analyses

The purity of complexes **1** and **2** was confirmed by X-ray power diffraction analyses, in which the experimental spectra of both compounds are almost consistent with their simulated spectra (Figs. S2 and S3).

Complexes **1** and **2** are air stable. The TG data of **1** shows that the complex is stable up to 163 °C; decomposition of **2** begins from 271 °C, which has higher thermal stability than **1**, as indicated in Fig. 3.

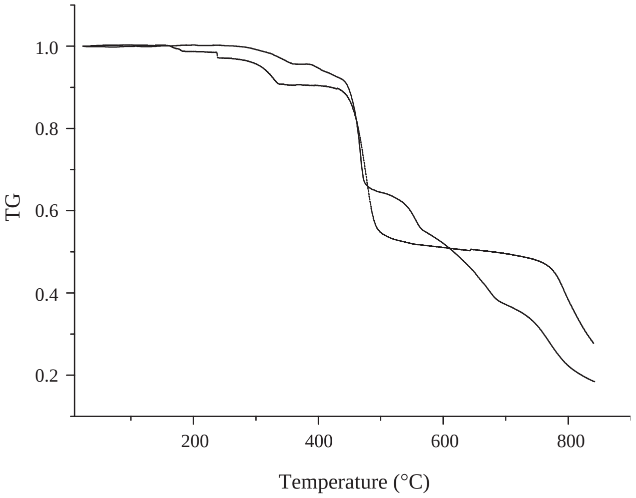

Fig. 3. TG curve of **1** (dashed line) and **2** (full line).

3.4. Photoluminescence properties

The emission spectra of complexes **1** and **2** in the solid state are investigated at room temperature. Excitation at 365 nm leads to strong blue-fluorescent emission band at 454 nm for **1** while intensely strong blue-fluorescent emission bands are observed at 442 and 480 nm for **2** under the same conditions, as indicated in Figs. 4 and 5. These emissions are neither metal-to-ligand charge transfer (MLCT) nor ligand-to-metal transfer (LMCT) in nature since the Zn^{2+} or Cd^{2+} ions are difficult to oxidize or to reduce due to their d^{10} configuration [23]. For the free H_2BDC ligand, the emission band at 387 nm ($\lambda_{ex} = 351$ nm) can be assigned to π–π^* transition. In addition, for the free H_2bibzim, the emission bands at 499, 535, 580 nm ($\lambda_{ex} = 491$ nm) are assigned to the intraligand $^1(\pi_2)$–$^1(\pi, \pi^*)$ transition [24]. Therefore, we assign the emissions described above for **1** and **2** to LLCT (ligand–ligand charge transfer) excited states. The shift of the emission bands for the two complexes is probably due to the differences of metal ions [25].

4. Supplementary materials

Fig. S1 illustrated a 3D framework for **2** and Figs. S2 and S3 indicated the experimental and simulated XRD spectra for both compounds are included. Crystallographic data (excluding structure factors) for the structure(s) reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication numbers: CCDC 259379 (**1**), CCDC 249725 (**2**). Copies of the data can be obtained free of charge on application to CCDC, 12, Union Road, Cam-

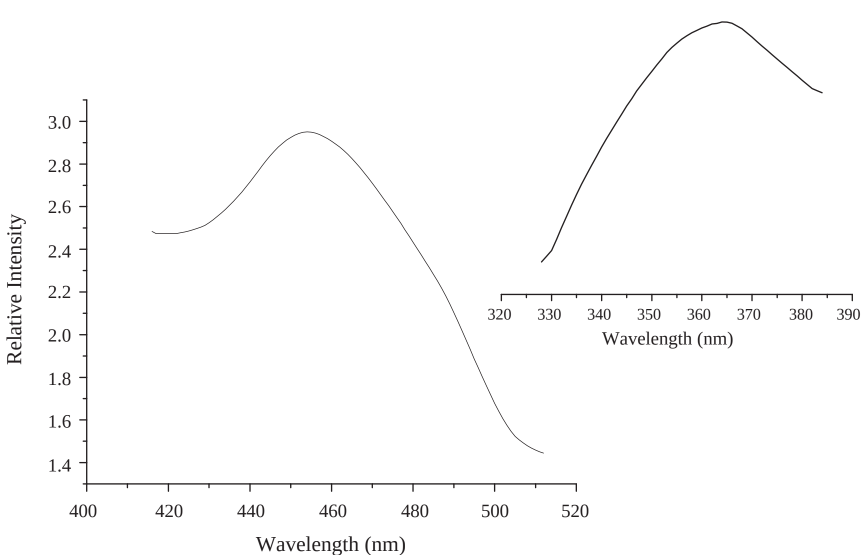

Fig. 4. Solid-state excitation and emission spectra of **1** at room temperature.

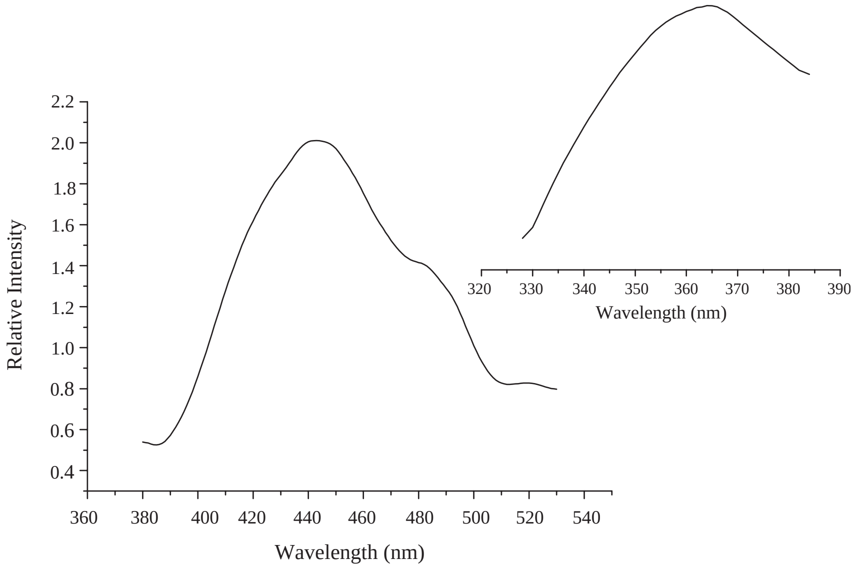

Fig. 5. Solid-state excitation and emission spectra of **2** at room temperature.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jssc.2005.07.035

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