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Syntheses, crystal structures, and properties of nickel and cadmium complexes containing imidazole derivatives

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Syntheses, crystal structures, and properties of nickel and cadmium complexes containing imidazole derivatives

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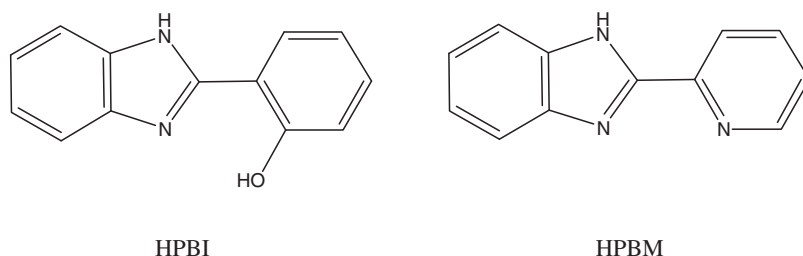
[Ni₃(C₁₃H₉N₂O)₅(CH₃OH)₂] (**1**) and [Cd(C₁₂H₈N₃)₃] (**2**) were synthesized in methanol and characterized by elemental analyses, IR spectra, and X-ray diffraction. Complex **1** is trinuclear with three unsymmetric Ni(II)'s having different coordinated environments bridged by four oxygen atoms from five ligands. Intermolecular antiferromagnetic interactions were observed in **1**. Complex **2** is mononuclear with Cd(II) six-coordinate by six nitrogen atoms from three bidentate HPBM anions. Complex **2** shows emission maxima in the blue region.

Keywords: Imidazole complexes; Crystal structure; UV-Vis spectrum; Magnetic properties; Fluorescence spectrum

1. Introduction

Complexes containing benzimidazole ligands have interesting topological frameworks and potential as function materials [1–8]. Metal complexes with 2-(2-hydroxyphenyl)benzimidazole derivatives have displayed good photoluminescence and magnetic properties [9–14]. Huang *et al.* reported bright red electroluminescent devices using europium complexes with 2-(2-pyridyl)benzimidazole [15]. Our group synthesized blue light-emitting pyridylbenzimidazole complexes of Cd(II) and Zn(II) [16]. Nickel imidazole complexes have an important role in biology, optical properties, and magnetic properties [17–19]. Photophysical properties of complexes are closely related to their structural features, so, it is important to prepare more imidazole complexes [20]. Continuing our research on benzimidazole ligands, herein we report the synthesis and characterization of nickel(**1**) and cadmium(**2**) compounds derived from 2-(2-hydroxyphenyl)benzimidazole (HPBI) and 2-(2-pyridyl)benzimidazole (HPBM) (scheme 1).

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Scheme 1. Illustration of the two ligands.

2. Experimental

2.1. Materials and methods

Salicylic acid, *o*-phenylenediamine and polyphosphoric acid (Aldrich) were used without purification. The other reagents and complexes were synthesized according to established procedures. UV-Vis spectra were recorded on a TU-1901 spectrometer. Fluorescence spectra of samples were recorded on a RF-5301 spectrometer. Elemental analyses were performed on a Perkin-Elmer 240c analyzer. IR spectra of samples were recorded on a MAGNA-560 spectrometer. The magnetic susceptibility of **1** was measured from 2 to 300 K with an applied field of 1000 Oe on a SQUID magnetometer using polycrystalline samples.

2.2. Syntheses

2.2.1. 2-(2-Hydroxyphenyl)benzimidazole (HPBI). A mixture of salicylic acid (2.5 g, 18.1 mmol) and *o*-phenylenediamine (1.95 g, 18.1 mmol) was stirred in concentrated polyphosphoric acid (30 g) at 200°C for 5 h. The hot mixture was poured into vigorously stirred cold water (200 mL). The pH of the mixture was adjusted to 7.0 by aqueous NaOH. The precipitate was filtered and then recrystallized from ethanol as white needle crystals (2.35 g, 61%).

2.2.2. Synthesis of 1. Solutions of 0.1 mmol $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ in 2 mL of methanol and 0.2 mmol HPBI in 20 mL methanol were mixed and then 1 mL triethylamine was added. The solution was refluxed for 3 h, filtered, and evaporated to dryness. The residue was dissolved in methanol. Crystals of **1** were obtained after three months. Anal. Calcd for $\text{C}_{67}\text{H}_{54}\text{N}_{10}\text{Ni}_3\text{O}_9$ (%): C, 60.94; H, 4.09; N, 10.61. Found (%): C, 61.03; H, 4.05; N, 10.58. IR (cm^{-1}): 1608, 1487, 1288, 467, 445.

2.2.3. Synthesis of 2. Solutions of 0.1 mmol $\text{Cd}(\text{Ac})_2 \cdot 2\text{H}_2\text{O}$ in 3 mL of methanol and 0.3 mmol HPBM in 20 mL methanol were mixed and then 1.5 mL triethylamine was added to the solution. The mixture was refluxed for 3 h. The precipitate was filtered off and the residue was dissolved in pyridine; crystals of **2** were obtained after 12 days. Anal. Calcd for $\text{C}_{36}\text{H}_{27}\text{CdN}_9\text{O}$ (%): C, 60.50; H, 3.78; N, 17.64. Found (%): C, 60.55; H, 3.78; N, 17.66. IR (cm^{-1}): 1610, 1500, 1475, 736, 367.

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

	1	2
Chemical formula	C ₆₇ H ₅₄ N ₁₀ Ni ₃ O ₉	C ₃₆ H ₂₇ CdN ₉ O
Temperature (K)	293(2)	293(2)
Wavelength (Å)	0.71069	0.71073
Formula weight	1319.33	714.1
Space group	<i>p</i> -1	<i>Pa</i> -3
Unit cell dimensions (Å, °)		
<i>a</i>	11.797(5)	18.8554(5)
<i>b</i>	14.688(5)	18.8554(5)
<i>c</i>	21.153(5)	18.8554(5)
α	96.992(5)	90.00
β	104.512(5)	90.00
γ	99.257(5)	90.00
Volume (Å ³), <i>Z</i>	3451(2), 2	6703.6(3), 80
Calculated density (Mg m ⁻³)	1.270	1.415
Absorption coefficient (mm ⁻¹)	0.869	0.695
<i>F</i> (000)	1364	2896
Crystal size (mm ³)	0.24 × 0.25 × 0.26	0.24 × 0.25 × 0.26
θ range for data collection (°)	1.01 to 28.32	1.87 to 28.31
Limiting indices	−11 ≤ <i>h</i> ≤ 15; −19 ≤ <i>k</i> ≤ 18; −28 ≤ <i>l</i> ≤ 19	−24 ≤ <i>h</i> ≤ 24; −13 ≤ <i>k</i> ≤ 24; −25 ≤ <i>l</i> ≤ 24
Reflections collected/unique	21,872/16072 [<i>R</i> (int) = 0.0361]	39,677/2778 [<i>R</i> (int) = 0.0739]
Completeness to θ	28.32(93.4%)	28.31(99.4%)
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents
Refinement method	Full-matrix least-squares on <i>F</i> ²	Full-matrix least-squares on <i>F</i> ²
Data/restraints/parameters	16,072/0/824	2778/0/146
Goodness-of-fit on <i>F</i> ²	0.917	0.921
Final <i>R</i> ₁ , <i>wR</i> ₂ [<i>I</i> > 2 σ (<i>I</i>)]	0.0768, 0.2457	0.0385, 0.0926
Final <i>R</i> ₁ , <i>wR</i> ₂ (all data)	0.1299, 0.2885	0.0897, 0.0973
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (eÅ ⁻³)	1.977 and −0.818	1.185 and −0.918

$$R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|; wR_2 = \sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]^{1/2}.$$

2.3. X-ray crystallography

Suitable single crystals of **1** and **2** were grown in methanol and pyridine, respectively. Diffraction data using ω scans with graphite monochromated Mo-K α radiation (0.71069) were collected at 293 K. Crystals were put in fine-focus sealed tube for data collection. A summary for crystal data and structure refinement of **1** and **2** is given in table 1. Selected bond lengths and angles are listed in tables 2–4. The data of all compounds were refined by full matrix least-squares on *F*² and structures were solved using SHELX 97 and refined with SHELX 97 [21].

3. Results and discussion

3.1. Structure description of **1**

Complex **1** is a trinuclear complex crystallizing in the triclinic space group *p*-1 with Ni(3)–Ni(2)–Ni(1) angles of 77.17(4)°. As shown in figure 1, the three unsymmetric Ni(II)'s with different coordinated environments are bridged by four oxygen atoms

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

N(1)–Ni(1)	1.998(5)	O(2)–Ni(2)	1.988(4)	O(5)–Ni(2)	2.191(4)
N(3)–Ni(2)	2.002(4)	O(3)–Ni(2)	2.044(4)	O(6)–Ni(3)	2.080(4)
N(5)–Ni(2)	2.017(5)	O(3)–Ni(1)	2.069(4)	O(7)–Ni(1)	2.092(5)
N(7)–Ni(3)	2.035(5)	O(3)–Ni(3)	2.300(4)	Ni(1)–Ni(2)	2.9121(12)
N(9)–Ni(3)	2.008(5)	O(4)–Ni(3)	1.996(4)	Ni(2)–Ni(3)	2.8549(11)
O(1)–Ni(1)	1.941(4)	O(4)–Ni(2)	2.028(4)		
O(2)–Ni(1)	1.975(4)	O(5)–Ni(3)	2.017(4)		

Table 3. Selected angles (°) for **1**.

O(1)–Ni(1)–O(2)	162.27(17)	N(3)–Ni(2)–O(3)	161.58(16)	O(4)–Ni(3)–O(5)	86.22(15)
O(1)–Ni(1)–N(1)	92.33(19)	N(5)–Ni(2)–O(3)	89.83(17)	N(9)–Ni(3)–O(5)	90.58(19)
O(2)–Ni(1)–N(1)	105.29(18)	O(4)–Ni(2)–O(3)	82.64(15)	O(4)–Ni(3)–N(7)	89.68(17)
O(1)–Ni(1)–O(3)	93.91(17)	O(4)–Ni(2)–O(5)	84.75(15)	N(9)–Ni(3)–N(7)	105.7(2)
O(2)–Ni(1)–O(3)	82.71(15)	N(3)–Ni(2)–O(5)	90.76(16)	O(5)–Ni(3)–N(7)	163.46(17)
N(1)–Ni(1)–O(3)	95.28(17)	N(5)–Ni(2)–O(5)	161.27(17)	O(4)–Ni(3)–O(6)	169.72(16)
O(1)–Ni(1)–O(7)	88.1(2)	O(4)–Ni(2)–O(5)	81.00(14)	N(9)–Ni(3)–O(6)	92.90(19)
O(2)–Ni(1)–O(7)	91.4(2)	O(3)–Ni(2)–O(5)	71.61(15)	O(5)–Ni(3)–O(6)	86.08(17)
N(1)–Ni(1)–O(7)	97.7(2)	O(2)–Ni(2)–Ni(3)	118.09(11)	N(7)–Ni(3)–O(6)	95.79(18)
O(3)–Ni(1)–O(7)	166.80(19)	N(3)–Ni(2)–Ni(3)	117.50(13)	O(4)–Ni(3)–O(3)	77.12(14)
O(1)–Ni(1)–Ni(2)	125.72(13)	N(5)–Ni(2)–Ni(3)	121.35(13)	N(9)–Ni(3)–O(3)	158.71(17)
O(2)–Ni(1)–Ni(2)	42.87(11)	O(4)–Ni(2)–Ni(3)	44.36(10)	O(5)–Ni(3)–O(3)	69.79(14)
N(1)–Ni(1)–Ni(2)	119.37(14)	O(3)–Ni(2)–Ni(3)	52.91(10)	N(7)–Ni(3)–O(3)	93.68(16)
O(3)–Ni(1)–Ni(2)	44.58(10)	O(5)–Ni(2)–Ni(3)	44.76(10)	O(6)–Ni(3)–O(3)	93.82(15)
O(7)–Ni(1)–Ni(2)	124.99(16)	O(2)–Ni(2)–Ni(1)	42.52(11)	O(4)–Ni(3)–Ni(2)	45.26(10)
O(2)–Ni(2)–N(3)	90.26(16)	N(3)–Ni(2)–Ni(1)	120.94(13)	N(9)–Ni(3)–Ni(2)	115.64(15)
O(2)–Ni(2)–N(5)	95.66(17)	N(5)–Ni(2)–Ni(1)	109.49(13)	O(5)–Ni(3)–Ni(2)	49.90(11)
N(3)–Ni(2)–N(5)	107.95(18)	O(4)–Ni(2)–Ni(1)	120.14(11)	N(7)–Ni(3)–Ni(2)	117.81(14)
O(2)–Ni(2)–O(4)	162.36(15)	O(3)–Ni(2)–Ni(1)	45.28(10)	O(6)–Ni(3)–Ni(2)	124.58(12)
N(3)–Ni(2)–O(4)	100.24(17)	O(5)–Ni(2)–Ni(1)	59.03(11)	O(3)–Ni(3)–Ni(2)	45.15(9)
N(5)–N(2)–O(4)	94.54(16)	Ni(3)–Ni(2)–Ni(1)	77.17(4)		
O(2)–Ni(2)–O(3)	83.04(15)	O(4)–Ni(3)–N(9)	93.98(18)		

Table 4. Selected bond lengths (Å) and angles (°) for **2**.

Cd(1)–N(1)	2.253(3)
Cd(1)–N(2)	2.488(3)
N(1)–Cd(1)–N(1)	105.47(8)
N(1)–Cd(1)–N(2)	71.26(10)
N(1)–Cd(1)–N(2)	156.57(9)
N(1)–Cd(1)–N(2)	97.64(9)
N(2)–Cd(1)–N(2)	86.00(10)

from five ligands. The Ni(1)···Ni(2) and Ni(2)···Ni(3) distances (2.9121(12) and 2.8549(11)) reveal that there is no direct metal–metal interaction [22]. Seven oxygen atoms involved in coordination come from methanol and HPBI; O(2), O(3) O(4), and O(5) are bridging. The Ni–N and Ni–O bond distances are 1.998–2.002 and 1.941–2.300 Å, respectively, comparable to literature values [23, 24].

We have reviewed the literature for Co(II), Mn(IV), Zn(II), and Cu(II) complexes of ligand HPBI [12, 14, 25]. Complexes of Co(II), Zn(II), and Cu(II) of HPBI are mononuclear with tetrahedral center ions coordinated by two deprotonated HPBI. The Mn(IV) complex of HPBI is binuclear with two six-coordinate manganese(IV) ions coordinated to two HPBI ligands and bridged by two oxides. Complex **1** is a new

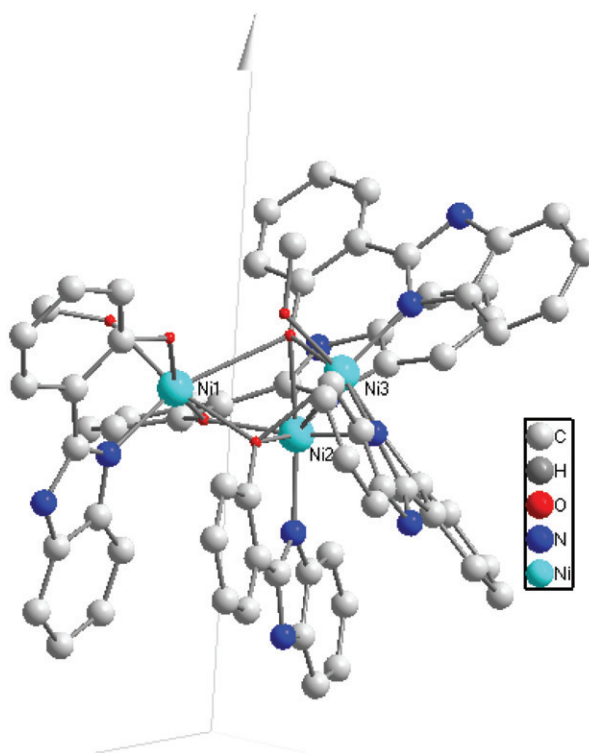


Figure 1. Molecule structure of **1**.

trinuclear complex, much more complicated, probably due to the relatively small ion radius creating hindrance and the presence of bridging oxygen atoms.

3.2. Structure description of **2**

As shown in figure 2, each Cd(II) is six-coordinate by six nitrogen atoms from the three bidentate HPBM anions. The coordination geometry of Cd(II) is distorted octahedral. Each deprotonated HPBM coordinates to one Cd(II) through pyridine N and imidazole N. The Cd–N (pyridine) distance of (2.488 Å) is longer than the Cd–N (imidazole) distance (2.253 Å).

In 2006, Yue and co-workers reported a Cd(II) complex of HPBM [Cd(L2)₂(H₂O)₂] with Cd(II) six-coordinate by four nitrogen atoms from the two bidentate HPBM anions and two water molecules [16]. There are no water molecules in **2**, which is a relatively stable structure.

3.3. Photophysical properties

The UV-Vis absorption spectra of HPBI, HPBM, **1**, and **2** were recorded at $1.0 \times 10^{-5} \text{ mol L}^{-1}$ in methanol at room temperature. The UV-Vis spectra of ligand HPBI and **1** are shown in “Supplementary material” section. The spectrum of **1** is similar with HPBI, but red-shifted 13 nm from formation of the complex altering the

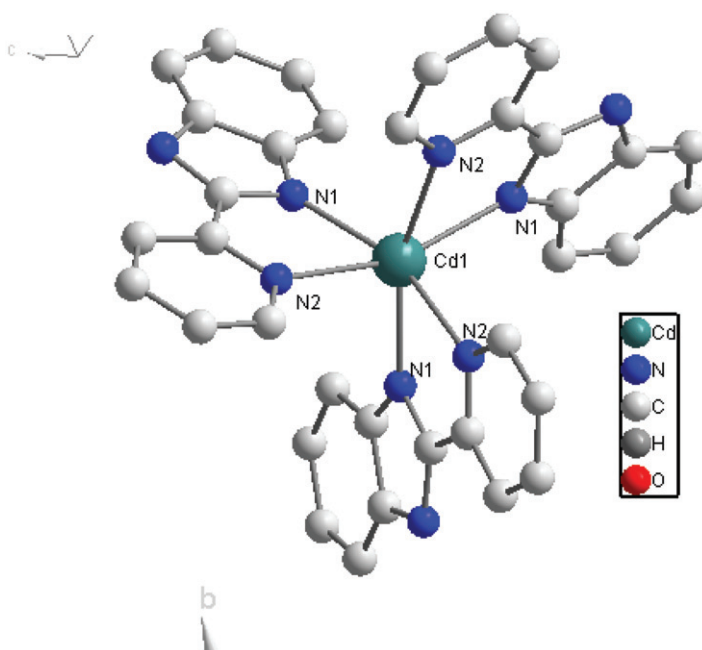


Figure 2. Molecule structure of **2**.

conjugated system. Compared with HPBI, the emissions of **1** blue shift (Supplementary material), indicating that in dilute solutions the intermolecular interactions between **1** are much weaker than between HPBI. The chelate structure may decrease the intermolecular interaction.

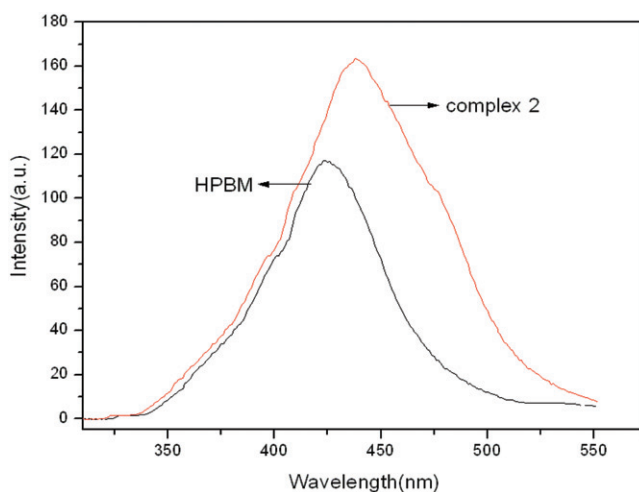
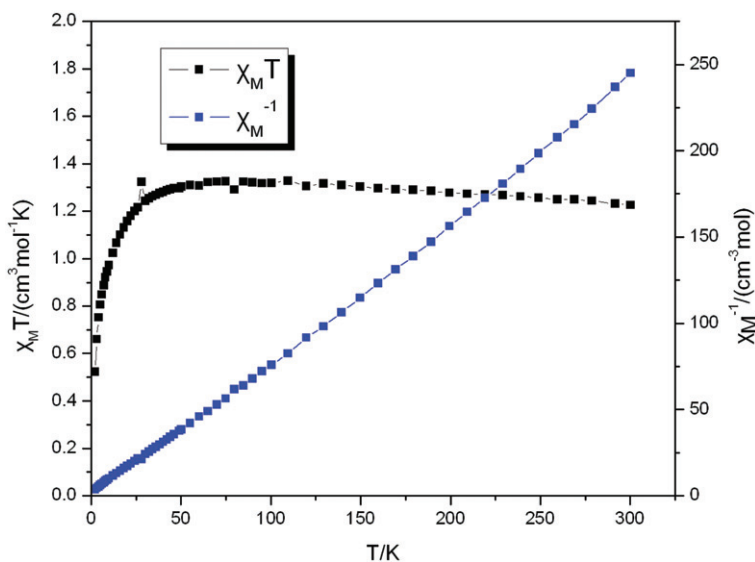
The UV-Vis spectra of ligand HPBM and **2** are also shown in “Supplementary material” section. The maximum absorptions of HPBM and **2** are 308 nm and 309 nm, assigned to intraligand $\pi-\pi^*$ transitions.

As shown in figure 3, the maximum emissions of HPBM and **2** are 418 nm and 435 nm, respectively, red-shifted for **2** compared to HPBM. This is assigned to $\pi-\pi^*$ fluorescence and Cd has no contribution to the light-emitting. The metals increase ligand rigidity, improving luminescent properties.

In 2006, Yue and co-workers researched the luminescent properties of $[\text{Cd}(\text{L}_2)_2(\text{H}_2\text{O})_2]$, which is a rare blue fluorescent material [16]. They reported the effect of water on zinc(II) and cadmium(II) complexes with pyridylimidazole by (DFT) B3LYP methods with coordinated water, resulting in low photoluminescent quantum efficiency [20]. Complex **2** has water replaced by HPBM allowing **2** to have improved luminescent properties. In future, **2** will be studied for electroluminescent display.

3.4. Magnetic properties

The $X_M T$ versus T curve of **1** suggests a weak intramolecular antiferromagnetic interaction between Ni(II) ions as shown in figure 4. The value of $X_M T$ at 300 K is $1.2257 \text{ cm}^3 \text{ mol}^{-1} \text{ K}$. When the temperature decreases, the $X_M T$ values remain basically unchanged until 50 K, when $X_M T$ values decrease rapidly reaching a value of

Figure 3. Emission spectra of HPBM and **2**.Figure 4. Plots of $X_M T$ vs. T and X_M^{-1} vs. T for **1**.

$0.5223 \text{ cm}^3 \text{ mol}^{-1} \text{ K}$ at 2 K. The magnetic data for **1** can be fitted to the Curie–Weiss law $X_M = C/(T - \theta)$, with $C = 1.474 \text{ cm}^3 \text{ mol}^{-1} \text{ K}$ and $\theta = -5.49 \text{ K}$. The negative θ value for **1** indicates antiferromagnetic interactions between Ni(II) ions.

4. Conclusion

Two new complexes, $[\text{Ni}_3(\text{C}_{13}\text{H}_9\text{N}_2\text{O})_5(\text{CH}_3\text{OH})_2]$ (**1**) and $[\text{Cd}(\text{C}_{12}\text{H}_8\text{N}_3)_3]$ (**2**), were synthesized in methanol and characterized. Complex **1** is trinuclear while **2** is

mononuclear. Compared with reported Zn(II), Cu(II), Co(II), and Mn(IV) complexes, the structures of **1** and **2** are more complicated. We reported magnetic properties of **1** and luminescent properties of **2**. Metal ions play significant roles in determining the structure and properties of these complexes.

Supplementary material

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Center, CCDC Nos 881784 and 881783 for **1** and **2**, respectively. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>).

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