APPLIED ORGANOMETALLIC CHEMISTRY
Appl. Organometal. Chem. 2007; **21**: 150–155
Published online in Wiley InterScience
(www.interscience.wiley.com) DOI:10.1002/aoc.1187

Two novel benzenedicarboxylate-metal complexes: synthesis, crystal structures and fluorescent properties

Yi-Shan Song[1], Bing Yan[1]* and Zhen-Xia Chen[2]

[1]Department of Chemistry, Tongji University, Shanghai 200092, People's Republic of China
[2]Department of Chemistry, Fudan University, Shanghai 200433, People's Republic of China

Received 18 October 2006; Revised 14 November 2006; Accepted 17 November 2006

Two novel benzenedicarboxylate–metal complexes, [Sm(nphth)(Hnphth)(H_2O)$_3$•H_2O]$_2$ and [Zn(nphth)(bipy)(H_2O) •H_2O]$_2$ (2) (H_2nphth = 3-nitrophthalic acid, bipy = 2,2′-bipyridine), have been synthesized under hydrothermal condition and characterized by single-crystal X-ray diffraction. Both complex 1 and 2 exhibit a dimeric structure, and nphth ligand shows different coordination mode in the f-block and d-block complexes. The fluorescent properties of two complexes are investigated; the results reveal that the two complexes show different kinds of fluorescence. Copyright © 2007 John Wiley & Sons, Ltd.

KEYWORDS: organic–metal complex; benzenedicarboxylic acid; crystal structure; fluorescent property

INTRODUCTION

Transition metal–organic frameworks (MOFs), which are diverse in structure and property, are currently attracting increasing attention not only owing to their fascinating applications in the areas of catalysis, co-operative magnetic behavior, nonlinear optical activity and electrical conductivity, but also because of their interesting topologies.[1–8] To exquisitely design the metal–organic frameworks from transition metals and organic ligands with novel architectures and desired functionalities by use of the principles of crystal engineering is one of the most challenging subjects in coordination chemistry today.[9,10]

The flexible benzenedicarboxylic acids are good candidates for the construction of novel metal–organic complexes, which show many important advantages than other organic ligands: they have two carboxyl groups that can completely or partially deprotonate, which reduces rich coordination modes; they can act not only as a hydrogen-bond acceptor but also as a hydrogen-bond donor, depending upon the number of deprotonated carboxyl groups; and they may connect metal ions in different directions due to their rigidity and polycarboxylate groups.

The metal–organic complexes of benzenedicarboxylic acids always show the high-dimensional structure. In order to obtain the one-dimensional complexes, one approach is to introduce terminal ligands such as 2,2′-bipyridine, phen or their derivatives into the carboxylate system because the terminal ligands reduce the available metal–ion binding sites to interdict polymer growth in other directions.[11,12] Many one-dimensional metal–organic polymers have been prepared by using mixed ligands of multicarboxylate and 2,2′-bipyridine, phen or their derivatives, in which the interdicting action of 2,2′-bipyridine, phen or their derivatives plays an important role in the formation of a one-dimensional chain.[11–17]

In addition, the hydrothermal method is effective for the crystal growth of many coordination polymers. Under hydrothermal conditions, the properties of the reactants and the interactions between organic and inorganic partners are quite different from those under conventional conditions in water. Therefore, various simple precursors and metastable compounds may be produced by hydrothermal reactions, and this facilitates crystal growth from solution.[18–20] Taking account of these, we chose the benzenedicarboxylate ligand and 2,2′-bipyridine as reactants to build metal architectures by a hydrothermal reaction. Interestingly, the results of single-crystal X-ray analysis reveal that complex **1** [Sm(nphth)(Hnphth)(H_2O)$_3$•H_2O]$_2$ and complex **2** [Zn(nphth)(bipy)(H_2O)•H_2O]$_2$ show a dimeric structure. In addition, two complexes show different kinds of fluorescence.

---

*Correspondence to: Bing Yan, Department of Chemistry, Tongji University, Shanghai 200092, People's Republic of China.
E-mail: byan@tongji.edu.cn
Contract/grant sponsor: National Natural Science Foundation of China; Contract/grant number: 20301013.

EXPERIMENTAL SECTION

Preparation of nphth–metal complexes

*Syntheses of [Sm(nphth)(Hnphth)(H_2O)$_3$·H_2O]$_2$ (**1**)*

$Sm(NO_3)_3 \cdot 6H_2O$ (0.3 mmol), H_2nphth (0.45 mmol) and bipy (0.3 mmol) were mixed in 15 ml deionized water. After stirring for 30 min, the mixture was placed in a 25 ml Teflon-lined reactor and heated at 160 °C in an oven for 3 days, then cooled slowly to room temperature; yellow column crystals of complex **1** suitable for X-ray diffraction analysis were obtained. Anal. calcd for $C_{16}H_{15}N_2O_{16}Sm$: C, 29 92; H, 2 34; N, 4.36%. Found: C, 30.05; H, 2 28; N, 4.42%. IR (KBr pellet, cm^{-1}): 1412 cm^{-1} (v_{sCOO-}), 1548 cm^{-1} (v_{asCOO-}).

*Synthesis of [Zn(nphth)(bipy)(H_2O) •H_2O]$_2$ (**2**)*

$Zn(CH_3COO)_2 \bullet 2H_2O$ (0.3 mmol), H_2nphth (0.3 mmol) and bipy (0.3 mmol) were mixed in 10 ml deionized water. After stirring for 30 min, the mixture was placed in a 25 ml Teflon-lined reactor and heated at 160 °C in an oven for 3 days, then cooled slowly to room temperature; brown column crystals of complex **2** suitable for X-ray diffraction analysis were obtained. Anal. calcd for $C_{18}H_{15}N_3O_8Zn$ (466.70): C, 46.28; H, 3.21; N, 9.00%. Found: C, 46.16; H, 3.27; N, 8.94%. IR spectrum exhibits a complicated pattern of bands in the range 4000–400 cm^{-1}: 1413 cm^{-1} (v_{sCOO^-}), 1541 cm^{-1} (v_{asCOO^-}).

Measurements and apparatus

Elemental analyses (C, H, N) were determined with an Elementar Cario EL elemental analyzer. IR spectra were recorded with a Nicolet Nexus 912 AO446 spectrophotometer (KBr pellet), 4000–400 cm^{-1} region. The luminescence (excitation and emission) spectra for the solid complex sample were determined with a Perkin-Elmer LS-55 spectrophotometer; whole excitation and emission slit widths were 10 and 5 nm, respectively.

Crystal structure determination

Diffraction data for a crystal with dimensions 0.25 × 0.20 × 0.15 mm for complex **1** and 0.15 × 0.10 × 0.10 mm for complex **2** were performed with graphite-monochromated Mo*Kα* radiation on a CCD area detector four-circle diffractometer, and were collected by the $\omega - 2\theta$ scan technique. The structures were solved by direct methods. All non-hydrogen atoms were refined anisotropically by full-matrix least-squares methods. The hydrogen atoms were added geometrically and not refined. All calculations were performed using SHELXS-97 and SHELXL-97.[21,22] A summary of crystallographic data and refinement parameters for complexes **1** and **2** are given in Table 1.

Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as a supplementary

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

Complex	Complex **1**	Complex **2**
Formula	$C_{16}H_{15}N_2O_{16}Sm$	$C_{18}H_{15}N_3O_8Zn$
Relative molecular weight *M*	641.65	466.70
Temperature	293(2) K	298(2) K
Wavelength	0.71073 Å	0.71073 Å
Crystal system	$P-1$	$P-1$
Space group	Triclinic	Triclinic
Unit dimensions	$a = 8.150(3)$ Å	$a = 7.5336(16)$ Å
	$b = 8.773(3)$ Å	$b = 10.442(2)$ Å
	$c = 15.122(5)$ Å	$c = 12.133(3)$ Å
	$\alpha = 100.219(4)°$	$\alpha = 88.677(3)°$
	$\beta = 91.182(4)°$	$\beta = 75.622(3)°$
	$\gamma = 104.507(4)°$	$\gamma = 77.571(3)°$
Volume	1027.7(6) Å^3	902.5(3) Å^3
Z	2	2
Calculated density	2.074 mg/m^3	1.717 mg/m^3
F(000)	630	476
Crystal size	0.25 × 0.20 × 0.15 mm	0.15 × 0.10 × 0.10 mm
θ range for data collection	2.44–25.01°	1.73–25.01°
Reflections/collected/unique	4350/3576 [R(int) = 0.0227]	3818/3143 [R(int) = 0.0287]
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	3576/12/348	3143/3/284
Goodness-of-fit on F^2	0.934	0.835
Final *R* indices [$I > 2\sigma(I)$]	$R1 = 0.0318, wR2 = 0.0613$	$R1 = 0.0428, wR2 = 0.0591$
Largest difference peak and hole	1.053 and −0.695 e. Å^{-3}	0.303 and −0.291 e Å^{-3}

DOI: 10.1002/aoc

publication (no. CCDC-276219 for complex **1** and CCDC-605448 for complex **2**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (Fax: +44 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

Crystal structure of complex 1

Complex **1** crystallizes in the triclinic system, with space group $P-1$. X-ray diffraction crystal structure analysis reveals that complex **1** forms a dimeric formulation $[Sm(nphth)(Hnphth)(H_2O)_3 \cdot H_2O]_2$ with two equivalent structural units corresponding to one-half of the dimer related by a crystallographic inversion center (Fig. 1). The two Sm centers are both equivalent in the same coordination environment and so have only one type of metal configuration. Each central samarium center is nine-coordinated completely by nine oxygen atoms, the coordination geometry of which can be described as a distorted monocapped square antiprismatic geometry. Among the above nine oxygen atoms, six are from the two coordinated H_2nphth ligands, the bond distances of between the samarium center and the carboxylate oxygen atoms being 2.393(3)–2.538(3) Å. It is worthy pointing out that the bond distance of Sm−O1 is shorter than Sm−O1,^{a} the reason being that the atom O1^{a} coordinated to the Sm(1) center forms a four-membered ring with oxygen atom O2^{a}, which enhances the reciprocity of the Sm center with an oxygen atom and lengthens the bond distance of Sm−O. In addition, the remaining three oxygen atoms from three water molecules coordinate the samarium center with the bond distances between the samarium center and the water oxygen atoms ranging from 2.374(4) to 2.508(4) Å. The bond average of Sm−O distance is 2.462 Å. In addition, the bong angles consisting of the Sm center and the carboxylic oxygen atoms are 51.19(10)° (O1^{a}−Sm−O2^{a}), 71.99(10)° (O1−Sm−O3) and 70.40(11)° (O7−Sm−O9), respectively.

In one unit of the complex **1**, there exist two H_2nphth anions. One (nphth) is dianionic, so the other one (Hnphth) must be monoprotonated to maintain electroneutrality. Correspondingly, two types of coordination modes of H_2nphth ligands exist in the structure: (1) the nphth anion acts as chelating-bridging tetradentate ligand towards the samarium centers with three carboxylic oxygen atoms—samarium centers are connected into a dimeric structure by this coordination [Fig. 2(a)]; and (2) the Hnphth anion acts as a chelating bidentate ligand towards samarium centers with two carboxylic oxygen atoms [Fig. 2(b)]. Selected bond lengths and angles for complexes **1** and **2** are listed in Table 2.

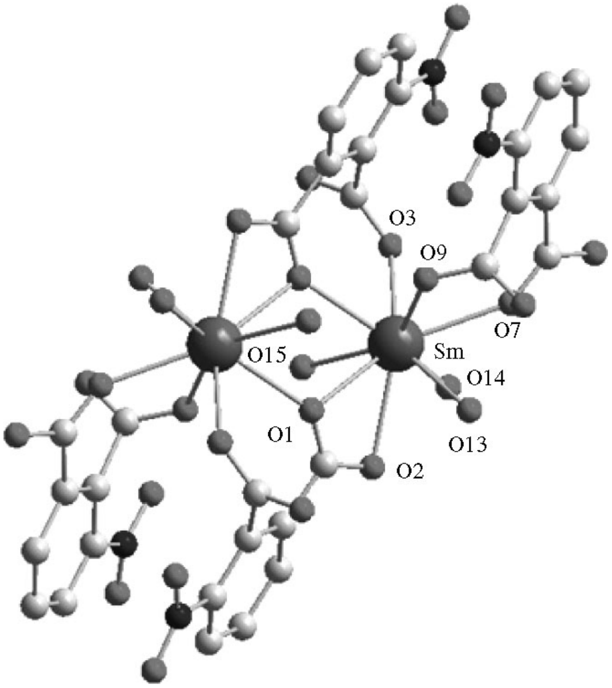
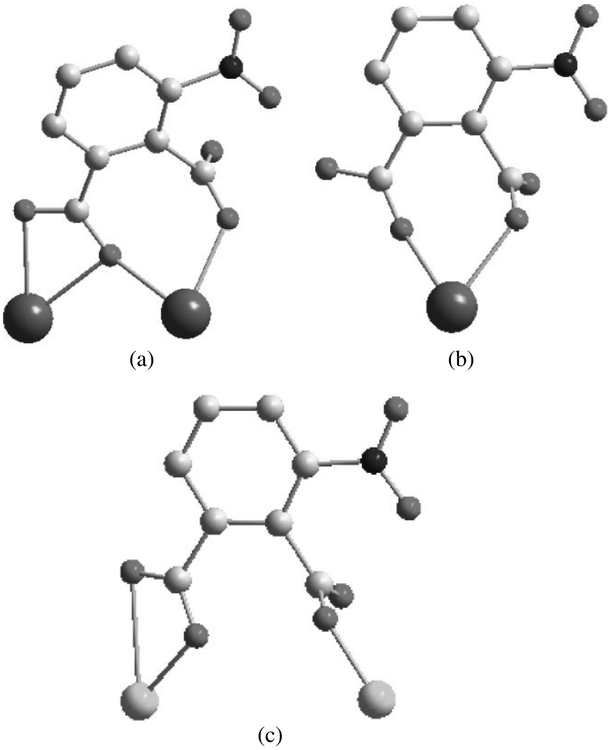


Figure 1. Molecular structure of complex **1**.


(a)

(b)

(c)

Figure 2. Coordination modes of nphth ligands in complexes **1** and **2**.

The existence of water molecules causes much hydrogen bonding in the structure of the title complex. There exist four types of O−H···O intermolecular hydrogen bonds: one is the O−H···O intermolecular hydrogen bonding between the oxygen atoms of the lattice water molecules and the oxygen atoms of the carboxylic groups, the bond angles of which are 168(7)° [O(16)−H(16B)···O(4)^{b}] and

Table 2. Selected bond distances (Å) and bond angles (deg) for complexes **1** and **2**

Sm(1)–O(13)	2.374(4)	Zn(1)–O(5)	1.990(2)
Sm(1)–O(3)	2.393(3)	Zn(1)–O(1)	2.044(2)
Sm(1)–O(7)	2.417(3)	Zn(1)–O(7)	2.079(3)
Sm(1)–O(15)	2.457(4)	Zn(1)–N(1)	2.139(3)
Sm(1)–O(9)	2.467(3)	Zn(1)–N(2)	2.141(3)
Sm(1)–O(1)	2.470(3)		
Sm(1)–O(14)	2.508(4)		
Sm(1)–O(2)[a]	2.534(3)		
Sm(1)–O(1)[a]	2.538(3)		
O(3)–Sm(1)–O(7)	91.32(11)	O(5)[a]–Zn(1)–O(1)	100.39(11)
O(13)–Sm(1)–O(15)	90.36(14)	O(5)[a]–Zn(1)–O(7)	89.18(12)
O(3)–Sm(1)–O(15)	143.60(12)	O(1)–Zn(1)–O(7)	93.83(11)
O(3)–Sm(1)–O(9)	73.44(11)	O(5)[a]–Zn(1)–N(1)	98.66(11)
O(7)–Sm(1)–O(9)	70.40(11)	O(1)–Zn(1)–N(1)	160.95(11)
O(3)–Sm(1)–O(1)	71.99(10)	O(7)–Zn(1)–N(1)	85.89(12)
O(7)–Sm(1)–O(1)	70.67(11)	O(5)[a]–Zn(1)–N(2)	116.39(11)
O(9)–Sm(1)–O(1)	126.30(11)	O(1)–Zn(1)–N(2)	96.20(11)
O(1)–Sm(1)–O(14)	71.96(12)	O(7)–Zn(1)–N(2)	150.12(12)
O(13)–Sm(1)–O(2)[a]	71.95(12)	N(1)–Zn(1)–N(2)	75.66(12)
O(3)–Sm(1)–O(2)[a]	140.97(11)		
O(7)–Sm(1)–O(2)[a]	127.40(11)		
O(9)–Sm(1)–O(2)[a]	120.04(11)		

Symmetry transformations used to generate equivalent atoms: for complex 1, [a] $-x$, $-y$, $-z$; for complex 2, [a] $-x+2$, $-y+1$, $-z+1$.

158(5)° [O(16) –H(16A)···O(8)[d]], respectively; the second is the intermolecular hydrogen bonding between the oxygen atoms of the coordinated water molecules and the oxygen atoms of the carboxylic groups, the bond angles of which are 171(6)° [O(13) –H(13A)···O(4)[c]], 160(5)° [O(14) –H(14A)···O(3)[c]], 137(5)° [O(14) –H(14B)···O(7)[a]] and 170(5)° [O(15) –H(15B)···O(c)[f]], respectively; the third is the intermolecular hydrogen bonding between the oxygen atoms of the coordinated water molecules and the oxygen atoms of the lattice water molecules, the bond angle of which is 162(5)° [O(13) –H(13B)···O(16)[e]]; and the last is the intermolecular hydrogen bonding between the oxygen atoms of the carboxylic groups and the oxygen atoms of the lattice water molecules, the bond angle of which is 179.6° [O(10) –H(10A)···O(16)[e]]. In particular, there exists one intramolecular hydrogen bonging in the structure between the oxygen atoms of the coordinated water molecules and the oxygen atoms of the carboxylic groups, the bond angle of which is 156(4)° [O(15) –H(15A)···O(8)]. All those hydrogen bonds link up the complex units, which results in a three-dimensional network and makes the whole network structure system stable. The detailed data of hydrogen bonding for complex **1** are shown in Table 3.

Crystal structure of complex 2

Complex **2** $[Zn(nphth)(bipy)(H_2O) \bullet H_2O]_2$ crystallizes in the triclinic system, with space group $P-1$. Figure 3 shows the fundamental asymmetrical unit of the complex **2**, which contains one zinc center, one nphth ligand, one coordinated water molecule and one lattice water molecule. Each zinc center is five-coordinated by three oxygen atoms and two nitrogen atoms, and the coordination geometry of zinc center can be described as a light-distorted quadrihedrogon geometry. Among above three oxygen atoms, two are from two different nphth ligands with a Zn–O1 distance of 2.044(2) Å and a Zn–O5 distance of 1.990(2) Å, respectively; the remaining one is from one coordinated water molecules with a Zn–O7 distance of 2.079(3) Å. In addition, the two nitrogen atoms are from the chelated bipy ligand with a Zn–N1 distance of 2.139(3) Å, a Zn–N2 distance of 2.141(3) Å and the angle of N1–Zn–N2 is 75.66(12)°.

All nphth molecules are protonated completely and taken part in the coordination to the zinc centers. There is only one coordination modes of nphth anions existing in the structure: the dinic anion acts as the tridentate-bridging ligand towards two zinc centers [Fig. 2(c)], and zinc centers are linked together by this coordination mode to from a dimeric structure. The dimers are further linked by strong O–H···O hydrogen bondings to generate a three-dimensional supramolecular architecture.

Fluorescent properties of complexes 1 and 2

Complexes **1** and **2** exhibit different kinds of fluorescence. The solid-state excitation spectrum of the complex **1** at room temperature shows that effective energy absorption mainly takes place in the narrow ultraviolet region of

Table 3. Hydrogen bonds (Å) for complex **1**

D–H···A	d(D–H)		d(H···A)	d(D···A)d(DHA)
O(16)–H(16B)···O(4)[b]	0.846(10)	1.902(19)	2.735(5)	168(7)
O(14)–H(14A)···O(3)[c]	0.843(10)	2.18(2)	2.987(5)	160(5)
O(16)–H(16A)···O(8)[d]	0.843(10)	1.95(2)	2.754(5)	158(5)
O(14)–H(14B)···O(7)[a]	0.841(10)	2.35(4)	3.019(5)	137(5)
O(13)–H(13A)···O(4)[c]	0.849(10)	1.804(15)	2.646(5)	171(6)
O(13)–H(13B)···O(16)[e]	0.848(10)	2.06(2)	2.873(5)	162(5)
O(15)–H(15B)···O(2)[f]	0.847(10)	1.916(15)	2.754(5)	170(5)
O(15)–H(15A)···O(8)	0.852(10)	1.978(19)	2.777(6)	156(4)
O(10)–H(10A)···O(16)[e]	0.82	1.77	2.587(6)	179.6

Symmetry transformations used to generate equivalent atoms: for complex **1**, [a] $-x, -y, -z$; [b] $-x+1, -y+1, -z+1$; [c] $-x+1, -y, -z$; [d] $-x, -y, -z+1$; [e] $-x+1, -y, -z+1$; [f] $x, y-z$.

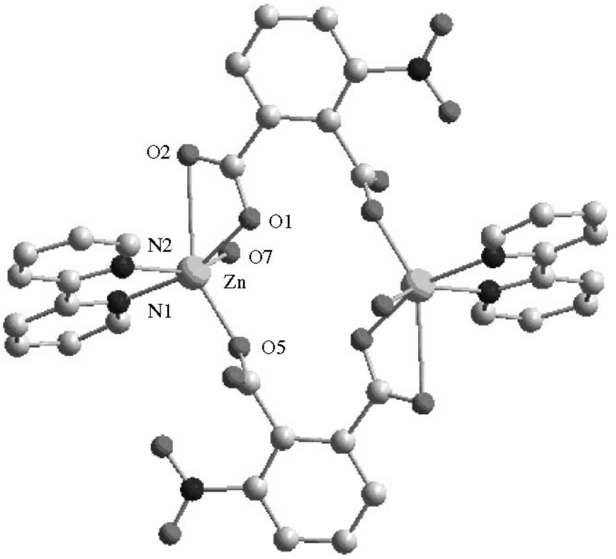


Figure 3. Molecular structure of complex **2**.

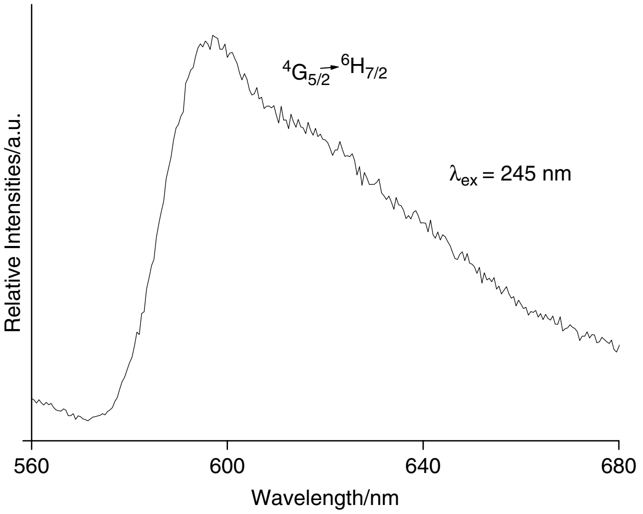


Figure 4. Luminescent spectra of complex **1**.

200–280 nm, and two main excitation peaks appear under orange emission of 596 nm: 216 and 245 nm, respectively. We further measured the corresponding emission spectra by selective excitation with the two different excitation wavelengths, and they show similar emission position except for little distinction of luminescent intensities. This indicates that two excitation bands are all the effective energy excitation for the luminescence of Sm^{3+} ion. On the contrary, we cannot obtain the characteristic emission of Sm^{3+} ion under the excitation of the wavelengths in the range 300–400 nm and it can be predicted that the excitation bands cannot produce the effective sensitization for the luminescence of Sm^{3+}. The emission spectrum of complex **1** (Fig. 4) shows one apparent emission bands under the excitation of 245 nm and the maximum emission wavelengths are at 597 nm, which is attributed to be the characteristic emission $^4G_{5/2} \rightarrow {}^6H_j$ ($j = 7/2$) transition of Sm^{3+} ion.

From the emission spectrum, complex **1** exhibits the weakest luminescence and the characteristic emission $^4G_{5/2} \rightarrow {}^6H_j$ ($j = 5/2$, $9/2$ and $11/2$) disappears. There are several reasons: the first is that there exist many internal energy levels ($^6F_{11/2}$, $^6F_{9/2}$, $\cdots\cdots$, $^6H_{11/2}$ etc) between the first excited state $^4G_{5/2}$ and the ground state $^6H_{9/2}$ of Sm^{3+}, which causes some non-radiative energy transfer process to lose the excited energy of nphth; the second is that the nphth ligand fluorescence emission and the energy transfer process from the lowest triplet state level of the ligand to the excited state levels of Sm^{3+} ions are two competitive processes, and the organic ligand with large conjugated structure may not be beneficial to the energy transfer from the ligand to the central metal ions; the third is that the samarium complex is apt to be deactivated by the solvent O–H vibrations.[23,24] The dominant luminescence mechanism for the solid Sm complex was given as luminescence from the triplet state of the ligand to the central samarium ion ($L^* - M$), namely the intramolecular energy transfer mechanism (LMET), as shown in Fig. 5.

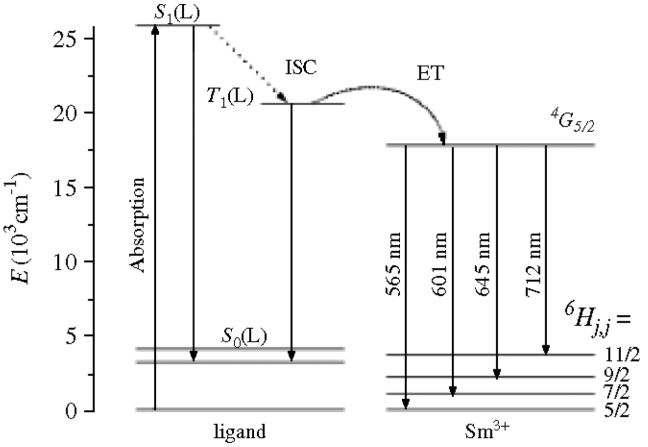


Figure 5. The sketch of intramolecular energy transfer mechanism in complex **1**.

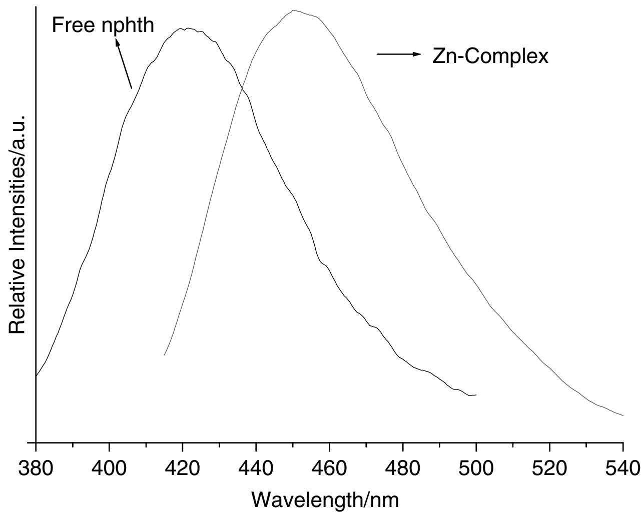


Figure 6. Luminescent spectra of complex **2** and free H_2nphth molecules.

Complex **2** exhibits a blue photoluminescence upon the radiation of ultraviolet light at room temperature. As shown in Fig. 6, the emission broad band of complex **2** mainly ranges from 400 to 550 nm with the maximum emission wavelengths at 450 nm. We further measured the emission spectrum of the free 3-nitrophthalic acid molecules, which shows one emission at 420 nm, indicating that blue luminescence can be observed when the nphth ligand is bound to Zn centers. The lower energy band would be assigned to the ligand-to-metal charge transfer, and the observed luminescence of complex **2** is attributed to the coordinated nphth ligand.[25]

Since hydrothermal products are usually stable and insoluble in common solvents arising from their polymeric structures, both complexes may be potential candidates for photoactive materials.

Acknowledgment

The authors gratefully acknowledged the financial support from the National Natural Science Foundation of China (20671072).

REFERENCES

1. Eddaoudi M, Kim J, Rosi N, Vodak D, Wachter J, O'Keefe M, Yaghi OM. *Science* 1996; **271**: 49.
2. Eddaoudi M, Li HL, Yaghi OM. *J. Am. Chem. Soc.* 1994; **116**: 1151.
3. Almeida Paz FA, Klinowski J. *Chem. Commun.* 2003; 1484.
4. Mahata P, Natarajan S. *Eur. J. Inorg. Chem.* 2005; 2156.
5. Long LS, Chen XM, Tong ML, Sun ZG, Ren YP, Huang RB, Zheng LS. *J. Chem. Soc., Dalton Trans.* 2001; 2888.
6. Hou Y, Wang ST, Shen EH, Wang EB, Xiao DR, Li YG, Xu L, Hu CW. *Inorg. Chim. Acta* 2004; **357**: 3155.
7. Kapoor P, Pathak A, Kapoor R, Venugopalan P. *Inorg. Chem.* 2002; **23**: 6153.
8. Batten SR, Robson R. *Angew. Chem., Int. Edn* 1998; **37**: 1460.
9. Eddaoudi M, Kim J, Rosi N, Vodak D, Wachter J, O'Keefe M, Yaghi OM. *Science* 2002; **295**: 469.
10. Kim J, Chen BL, Reineke TM, Li HL, Eddaoudi M, Moler DB, O'Keefe M, Yaghi OM. *J. Am. Chem. Soc.* 2001; **29**: 8239.
11. Li XJ, Sun DF, Cao R, Sun YQ, Wang YQ, Bi WH, Gao SY, Hong MC. *Inorg.Chem. Commun.* 2003; **6**: 908.
12. Song YS, Yan B, Chen ZX. *Appl. Organometal. Chem.* 2006; **20**: 44.
13. Oshio H, Ichida H. *J. Phys. Chem.* 1995; **99**: 3294.
14. Cotton FA, Daniels LM, Lin C, Murillo CA. *Inorg.Chem. Commun.* 2001; **4**: 130.
15. Castillo O, Luque A, Iglesias S, Guzman-Miralles C, Roman P. *Inorg. Chem. Commun.* 2001; **4**: 640.
16. Moghimi A, Alizadeh R, Shokrollahi A, Aghabozorg H, Shamsipur M, Shockravi A. *Inorg. Chem.* 2003; **42**: 1616.
17. Plater MJ, Foreman MRSJ, Howie RA, Skakle JMS, Hursthouse MB. *Inorg. Chim. Acta* 2001; **319**: 159.
18. Ruf M, Durfee WS, Pierpont CG. *Chem. Commun.* 2004; 1022.
19. Zhang LZ, Cheng P. *J. Chem. Phys.* 2003; **119**: 13061.
20. Wang YB, Zheng XJ, Zhuang WJ, Jin LP. *Eur. J. Inorg. Chem.* 2003; 3572.
21. Sheldrick GM. *Acta Crystallogr.* 1990; **A46**: 467.
22. Sheldrick GM. *SHELXS-97, a Program for X-Ray Crystal Structure Solution, and SHELXL-97, a Program for X-ray Structure Refinement.* Gottingen University: Gottingen, 1997.
23. Yan B, Song YS. *J. Fluor.* 2004; **14**: 289.
24. An BL, Gong ML, Li MX, Zhang JM. *J. Mol. Struct.* 2004; **687**: 1.
25. Yang WY, Schmider H, Wu QG, Zhang YS, Wang SN. *Inorg. Chem.* 2000; **39**: 2397.

DOI: 10.1002/aoc