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# Investigations into the synthesis and fluorescence properties of Tb(III) complexes of a novel bis-β-diketone-type ligand and a novel bispyrazole ligand

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## Abstract

A novel bis- $\beta$ -diketone organic ligand, 1,1'-(2,6-bispyridyl)bis-3-(*p*-methoxyphenyl)-1,3-propanedione (L<sub>1</sub>) and its derivatives, a novel bispyrazole ligand, 2,6-bis(5-(4-methoxyphenyl)-1H-pyrazol-3-yl)pyridine (L<sub>2</sub>) were designed and synthesized and their complexes with Tb(III) ion were successfully prepared. The ligands and the corresponding metal complexes were characterized by elemental analysis, infrared, proton nuclear magnetic resonance spectroscopy and TG-DTA. Analysis of the IR spectra suggested that the lanthanide metal ion Tb(III) coordinated to the ligands via the nitrogen atom of the pyridine ring and the carbonyl oxygen atoms for ligand L<sub>1</sub> and the nitrogen atom of the pyrazole ring for ligand L<sub>2</sub>. The fluorescence properties of the two complexes in solid state were investigated and it was discovered that the Tb(III) ions could be sensitized by both the ligand (L<sub>1</sub>) and ligand (L<sub>2</sub>) to some extent. In particular, the complex of ligand (L<sub>2</sub>) is a better green luminescent material that could be used as a candidate material in organic light-emitting devices (OLEDs) since it could be much better sensitized by the ligand (L<sub>2</sub>), and the fluorescence intensity of Tb(III) complex of L<sub>2</sub> are almost as twice strong as L<sub>1</sub>'s. © 2007 Elsevier B.V. All rights reserved.

Keywords: Bis-B-diketone; Bispyrazole; Synthesis; Fluorescence; IR spectra; Lanthanide complexes

# 1. Introduction

Organic electroluminescence (OEL) has been studied extensively for its applications because of its low drive voltage, suitability for integrated circuit and potential application for large flat panel display [1,2]. As some lanthanide ions, e.g. Eu<sup>3+</sup> and Tb<sup>3+</sup>, possess good luminescence characteristics (high color purity) based on the transitions between the 4f energy levels, a series of compounds activated Eu<sup>3+</sup> and Tb<sup>3+</sup> have been studied for practical application as phosphors and laser materials [3,4]. Terbium(III) has four narrow emission bands corresponding to the  ${}^{5}D_{4} \rightarrow {}^{7}F_{j}$  transitions, where j=6, 5, 4, 3. The strongest transition,  ${}^{5}D_{4} \rightarrow {}^{7}F_{5}$ , occurs at approximately 544 nm, the narrow intense green terbium luminescence [5]. However, several ternary rare earth complexes applied to prepare OEL devices showed weak visible emission and low electroluminescence efficiency [6,7]. In principle, electroneutral metal complexes may

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form uniform thin film in vacuum vapor deposition and are reasonably stable to heat, which is required for OEL displays [8]. Further investigations into the relationship between the structures of organic ligands and the energy levels of lanthanide ions will give evidences for designing high luminescent lanthanide organic complexes.

Since the 1960s, the complexes of Eu(TTA)<sub>3</sub>Phen, Sm(TTA)<sub>3</sub>Phen have attracted considerable attention because of their high fluorescence emission efficiency, which owes to high absorption coefficient of HTTA and the synergistic effect of 1,10-phenanthroline [9,10]. The complexes of Eu(III), Tb(III), Sm(III) and Gd(III)ions with  $\pi$ -conjugated ligands such as  $\beta$ -diketones have also been examined as emitting materials for OLEDs [11] following the reports by Okamoto and his co-workers. Yan and Song put more emphasis on the Eu<sup>3+</sup>, Tb<sup>3+</sup>, Sm<sup>3+</sup>, Dy<sup>3+</sup> and Gd<sup>3+</sup> complexes with 2,2'-bipyridine-*N,N'*-dioxide [12], 5-bromonicotinic acid [13], 2,6-pyridine dicarboxylic acid and phthalate, [14,15], etc., the corresponding coordination behavior and symmetry of central metal ions were investigated, and the outstanding luminescence properties were discussed especially. However, the quantum efficiency of

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most of these complexes is unfortunately still low. Chemists have realized that it is essential to design ligands, which have better energy transfer property to the lanthanide metal ion.

In our earlier work, we have designed and synthesized a novel, pyridine-containing, bis- $\beta$ -diketone-type ligand, 1,1'-(2,6-bispyridyl)bis-3-phenyl-1,3-propanedione, and studied the fluorescence properties of its Eu(III), Tb(III), Sm(III) and Gd(III) complexes in the solid state [16]. The result showed that the complexes with Tb(III) exhibited an strong green luminescent, but the quantum efficiency of these complexes are not high enough. In an attempt to increase the intensity of the lanthanide fluorescence exploiting the antenna effect, in this paper, on the basis of 1,1'-(2,6-bispyridyl)bis-3-phenyl-1,3-propanedione, another novel bis-β-diketone organic ligand, 1,1'-(2,6-bispyridyl)bis-3-(pmethoxyphenyl)-1,3-propanedione  $(L_1)$ , and its derivative, bispyrazole ligand, 2,6-bis(5-(4-methoxyphenyl)-1H-pyrazol-3-yl) pyridine (L<sub>2</sub>) were synthesized (Scheme 1), the corresponding terbium complexes were synthesized, characterized and the luminescence properties for the two terbium complexes were investigated as well.

## 2. Experimental

## 2.1. Materials

Pyridine-2,6-dicarboxylic acid and other reagents used were purchased and used as analytical grade. Rare earth chlorides were prepared according to the literature method [17] and [18].

#### 2.2. Methods

Contents of carbon, hydrogen and nitrogen were determined using an Elementar vario EL elemental analyzer. Content of Tb(III) was determined by EDTA titration. Infrared spectra (4000–400 cm<sup>-1</sup>) were recorded with samples as KBr discussing a Nicolet NEXUS 670 FTIR spectrophotometer. <sup>1</sup>H NMR spectra was measured by using a Bruker-400 MHz nuclear magnetic resonance spectrometer with CDCl<sub>3</sub> or DMSO as solvents and TMS as internal reference. Differential thermal analysis (DTA) was carried out using a Shimadzu DT-40. Fluorescence measurements were made on a Hitich F-4500 spectrometer.

## 2.3. Synthesis of the ligands

2.3.1. Synthesis of 1, l'-(2,6-bispyridyl)bis-3-(pmethoxyphenyl)-1,3-propanedione ( $L_1$ )

To a suspension of freshly cut sodium (1.0 g, 44.0 mmol)in anhydrous toluene (50.0 ml) was placed in a three-necked, round-bottom flask, fitted with a condenser, heated up with silicone oil till the sodium was molten. The mixture was then cooled to  $110 \,^{\circ}$ C and dimethyl 2,6-pyridinedicarboxylate(4.3 g 22.0 mmol) wad added into the mixture. The mixture in the flask was stirred, meanwhile, a solution of *p*-methoxy acetophenone (6.6 g, 44.0 mmol) in toluene (25.0 ml) was added dropwise. The reaction mixture was incubated and stirred at the temperature of  $110 \,^{\circ}$ C for about 2 h until the dark yellow sodium salt precipitated. The sodium salt was collected by filtration, washed thoroughly with petroleum ether and dried. The dried solid was



Scheme 1. Synthesis of the ligands (L1 and L2).

Table 1	
Elemental analytical	data for the complexes

Complex	Tb (%) found (calc.)	C (%) found (calc.)	H (%) found (calc.)	N (%) found (calc.)
$\frac{Tb_{2}(L_{1})_{3}\cdot 5H_{2}O}{Tb(L_{2})_{3}\cdot 4H_{2}O}$	18.71 (18.69)	53.10 (52.92)	3.98 (4.29)	2.62 (2.47)
	10.76 (10.60)	60.13 (60.00)	4.58 (4.73)	14.36 (14.00)

added to dilute hydrochloric acid (2.0 M, 50.0 ml) and the resulting precipitate was collected by filtration. The crude products were recrystallized from methanol to give the ligand  $(L_1)$  (8.1 g, 85.5%).

Physical properties of  $L_1$ : The <sup>1</sup>H NMR spectrum shows the presence of several keto-enol tautomers. In this paper, we described only the major tautomer, the bis(keto-enol) system, which formed yellow microcrystals from methanol, mp 220–221 °C; IR (KBr): v<sub>max</sub>(cm<sup>-1</sup>); 3422 (C=C-OH), 2924 (CH<sub>2</sub>), 2845 (-OCH<sub>3</sub>), 1616 (C=C), 1570 (C=N), 1533 (C=C), 1250 (Ar–O), 833 (Phen); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ16.5 (d, 2H, OH), 8.05-8.35 (m, 3H, Py), 7.04-7.45 (m, 8H, Ph), 3.92 (s, 6H, OCH<sub>3</sub>); Elemental analytical (calc.) C%69.70 (69.62), H% 4.92 (4.87), N% 3.43 (3.25).

#### 2.3.2. Synthesis of

# 2,6-bis(5-(p-methoxyphenyl)-1-H-pyrazol-3-yl) pyridine $(L_2)$

Hydrazine hydrate (4.0 ml, 85.0%) was added dropwise to a solution of 1,1'-(2,6-bispyridyl)bis-3-(p-methoxyphenyl)-1,3propanedione (1.72 g, 40.0 mmol) in acetic acid (50.0 ml) under stirring at room temperature for 30 min, The mixture was heated to 60 °C and incubated for about 4 h until the precipitate vanished, then was cooled to room temperature and ice water (20.0 g)was added to the mixture, a white precipitate formed. The precipitate was collected by filtration and washed with water. Recrystallization from acetone gave the ligand  $(L_2)(1.1 \text{ g})$ 65%). mp 240–241 °C. IR (KBr):  $\nu_{max}$ (cm<sup>-1</sup>): 3270 (N–H), 2835 (-OCH<sub>3</sub>), 1675, 1582, 1529, 1466, 1315 (skeleton of Ph, Py and Pyraz), 1248 (C–N), <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, ppm):  $\delta$ 13.45 (s, 2H, NH), 8.05–7.82 (m, 3H, Py), 7.05–7.23 (m, 8H, C<sub>6</sub>H<sub>5</sub>), 6.05 (s, 2H, Pyraz.), 3.06 (s, 6H, OCH<sub>3</sub>); Elemental analytical (calc.) C% 71.08 (70.92), H% 4.82 (4.96), N% 16.84 (16.55).

## 2.4. Synthesis of the complexes

The ligand  $L_1$  (3.0 mmol, 1.293 g)/ $L_2$  (6.0 mmol, 2.538 g) and the TbCl<sub>3</sub>·6H<sub>2</sub>O (2.0 mmol) were added to the ethanol (50.0 ml). The mixtures were stirred at 60 °C for 24 h at room temperature. A yellow/white compounds precipitated, the Tb(III) complexes was separated from the solution by suction filtration, purified by washing for several times with ethanol and chloroform and dried for 24 h in a vacuum to give a flaky solid, the complex of  $L_1$  is yellow power and the complex of  $L_2$  is white power.

# 3. Result and discussion

## 3.1. Properties of the complexes

The results of elemental data for the complexes presented in Table 1 indicated that the composition of the complexes conforms to  $Tb_2(L_1)_3 \cdot 5H_2O$ , and  $Tb(L_2)_3 \cdot 4H_2O$ , respectively. The both complexes were found to be soluble in H<sub>2</sub>O, DMF, DMSO and acetone, slightly soluble ethanol, and Tb<sub>2</sub>(L<sub>1</sub>)<sub>3</sub>·5H<sub>2</sub>O soluble in CHCl<sub>3</sub> and methanol, but  $Tb(L_2)_3 \cdot 4H_2O$  slightly soluble in methanol and CHCl<sub>3</sub>, they were both insoluble in benzene, diethyl ether and tetrahydrofuran, and may be kept in air for a long time.

## 3.2. IR spectra

Table 2 describes the characteristic bands of ligands and their complexes. The  $\nu$ (C=O),  $\nu$ (C=C) and  $\nu_{pyridine ring}$  (C=N) vibrations of the free ligand (L<sub>1</sub>) are at 1616, 1533 and  $1570 \text{cm}^{-1}$ , The band at 3422 in the free ligand  $(L_1)$  can be assigned to  $\nu$ (=C-OH). In the complex of Tb<sub>2</sub>(L<sub>1</sub>)<sub>3</sub>·5H<sub>2</sub>O, these bands are shifted downfield by  $10 \text{ cm}^{-1}$  for  $\nu$ (C=O) and 15 cm<sup>-1</sup> for  $\nu$ (C=C) and 21 cm<sup>-1</sup> for  $\nu$ (=C-OH) and shifted upfield by 65 cm<sup>-1</sup> for  $\nu$ (C=N). The  $\nu$ (N–H),  $\nu$ <sub>pyridine ring</sub> (C=N) and  $\nu$ <sub>pyraz ring</sub> (C-N) vibrations of the free ligand (L<sub>2</sub>) are at 3270, 1582 and  $1248 \text{ cm}^{-1}$ . In the complex, these bands are shifted downfield by 66 cm<sup>-1</sup> for  $\nu$ (N–H) and shifted upfield by 77 cm<sup>-1</sup> for  $v_{\text{pyridine ring}}$  (C=N) and 88 cm<sup>-1</sup> for  $v_{\text{pyraz ring}}$  (C-N). All the shifts indicate that carbonyl oxygen atoms, pyridine nitrogen atoms and pyrazole nitrogen atoms take part in coordination.

The absorption bands assigned to the coordinated Tb-O and Tb-N were observed at about 431 and  $530 \text{ cm}^{-1}$  for the complexes respectively.

The results of elemental analysis and IR spectroscopy indicate that coordination only involves the enol-forms of the ligand (L<sub>1</sub>).

Table	2	
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Characteristic IR	bands (cm <sup>-1</sup> )	) of the	ligands and	their co	mplexes

Compounds	νС–ОН	vN–H	v-OCH3	νC=Ο	νC=N	vC=C	vC–N	vO–Ar	vTb–O	vTb–N
L <sub>1</sub>	3422		2845	1616	1570	1533		1250		
L <sub>2</sub>		3270	2835		1582		1248	1268		
$Tb_2(L_1)_3 \cdot 5H_2O$	3401		2841	1606	1635	1518		1255	431	
$Tb(L_2)_3{\cdot}4H_2O$		3204	2833		1659		1336	1278		530



#### 3.3. Thermogravimetric analysis

In order to examine the thermal stability of the complexes, thermal gravimetric (TG) and differential thermal analyses (DTA) were carried out between 30 to 700°C in the static atmosphere of air (Fig. 1). The complex of  $Tb_2$  (L<sub>1</sub>)<sub>3</sub>·5H<sub>2</sub>O is stable up to  $450 \,^{\circ}$ C at which temperature it begins to melt. The TG curve of  $Tb_2(L_1)_3 \cdot 5H_2O$  indicates the release of 5.0 water molecules up to 120 °C (observed 5.10%, calc. 5.29%) to give the product of water-free  $Tb_2(L_1)_3$ , which is stable up to 450 °C (Fig. 1). The weight loss of 75.21% (calc. 76.05%) from 450 to 650 °C is equivalent to the loss of 3 coordinated ligands (L1). The solid residue formed at around 650 °C is suggested to be Tb<sub>2</sub>O<sub>3</sub>. Similarly, the complex of Tb( $L_2$ )<sub>3</sub>·4H<sub>2</sub>O is stable up to 460 °C at which temperature it begins to decompose. The TG curve of Tb( $L_2$ )<sub>3</sub>·4H<sub>2</sub>O indicates the release of ligands( $L_2$ ) which takes place at 460 °C (observed 83.23%, calc. 84.60%). The solid residue formed at around 560 °C is suggested to be Tb<sub>2</sub>O<sub>3</sub>.

## 3.4. Fluorescence studies

The solid complexes have characteristics line emission of f–f transition of metal ions when they are excited by UV light. The fluorescence characteristics of the complexes in solid state are listed in Table 3. The maximum excitation wavelengths ( $\lambda_{ex}$ ) of the Tb<sub>2</sub>(L<sub>1</sub>)<sub>3</sub>·5H<sub>2</sub>O and Tb(L<sub>2</sub>)<sub>3</sub>·4H<sub>2</sub>O complexes were 271 and 282 nm, respectively. Fluorescent spectra for the Tb(III) com-

Table 3	
Fluorescence data of the complexes at room temperature	



Fig. 2. The excitation and emission spectrum of the complex of  $Tb_2(L_1)_3 \cdot 4H_2O$ . The excitation and emission slit widths were 2.5 nm in solid state and the drive voltage was 400.0 V.

plexes were measured at the drive voltage of 400 V. The emission spectrums of the solid Tb<sup>3+</sup> complexes consist of four main lines at approximate 493 nm ( ${}^{5}D_{4} \rightarrow {}^{7}F_{6}$ ), 545 nm ( ${}^{5}D_{4} \rightarrow {}^{7}F_{5}$ ), 584 nm ( ${}^{5}D_{4} \rightarrow {}^{7}F_{4}$ ), and 621 nm ( ${}^{5}D_{4} \rightarrow {}^{7}F_{3}$ ) (Fig. 1). Due to the presence of a scattering signal near 490 nm, the peak height at 545 nm for terbium was used to measure the fluorescence intensities. We can see that the emission band ( ${}^{5}D_{4} \rightarrow {}^{7}F_{5}$ ) is obviously stronger than the other emission bands ( ${}^{5}D_{4} \rightarrow {}^{7}F_{6}$ ,  ${}^{5}D_{4} \rightarrow {}^{7}F_{4}$ ,  ${}^{5}D_{4} \rightarrow {}^{7}F_{3}$ ) from the Figs. 2 and 3.

According to the literature [16], we knew that very strong fluorescence could be observed from the Tb(III) complex. Comparing the complexes of  $Tb_2(L_1)_3 \cdot 5H_2O$ ,  $Tb(L_2)_3 \cdot 4H_2O$  and the Tb(III) complexes of 1,1'-(2,6-bispyridyl)bis-3-phenyl-1,3propane-dione [16] under the same experimental conditions, we can see that the fluorescence intensity observed from Tb(L<sub>2</sub>)<sub>3</sub>·4H<sub>2</sub>O are the strongest and is twice as strong as that of  $Tb_2(L_1)_3 \cdot 5H_2O$ , and the intensity of  $Tb_2(L_1)_3 \cdot 5H_2O$ is much stronger than that of the Tb(III) complexes of 1,1'-(2,6-bispyridyl)bis-3-phenyl-1,3-propane-dione. It appears that the energy-transfer from the organic ligand  $(L_2)$  to the central Tb(III) ions is the most effective compared to the ligand (L<sub>1</sub>) and ligand, 1,1'-(2,6-bispyridyl)bis-3-phenyl-1,3-propanedione that we have reported[16], which could be attributed to the following factors. Firstly, the ligand L<sub>1</sub> has two activating groups (-OCH<sub>3</sub>) which donate electrons to the aromatic rings

Complexes	State	Slit (nm)	$\lambda_{ex}$ (nm)	$\lambda_{em}$ (nm)	RFI <sup>a</sup>	Assignment
$\overline{\text{Tb}_2(\text{L}_1)_3\cdot\text{5H}_2\text{O}}$	Solid	2.5	282	493	2245	$^5D_4 \rightarrow {}^7F_6$
				545	4774	$^{5}D_{4} \rightarrow ^{7}F_{5}$
				584	435	$^{5}D_{4} \rightarrow ^{7}F_{4}$
				623	118	$^5D_4 \rightarrow {}^7F_3$
$Tb(L_2)_3{\cdot} 4H_2O$	Solid	2.5	271	492	1311	${}^{5}\text{D}_{4} \rightarrow {}^{7}\text{F}_{6}$
				547	9135	$^{5}\text{D}_{4} \rightarrow ^{7}\text{F}_{5}$
				586	261	$^{5}D_{4} \rightarrow ^{7}F_{4}$
				622	113	$^5D_4 \rightarrow {}^7F_3$



Fig. 3. The excitation and emission spectrum of the complex of  $Tb(L_2)_3 \cdot 5H_2O$ . The excitation and emission slit widths were 2.5 nm in solid state and the drive voltage was 400.0 V.

and enlarges the ligands'  $\pi$ -congjugated system; secondly, the symmetry alteration of the Tb<sup>3+</sup> ions in the complex might be beneficial to the energy transfer in the solid terbium complex; thirdly, the good rigid planar structure of the terbium complex was formed by the ligand (L<sub>2</sub>), which could better sensitize the luminescence; fourthly, the lengths of Tb–N were shorter than those of Tb–O, and the decrease of distances between the central Tb<sup>3+</sup> ions and donor nitrogen atoms from the pyrazole groups were beneficial to the energy transfer from the ligands to the central ions.

## 4. Conclusion

We have successfully synthesized a novel bis- $\beta$ -diketo-type and a novel bis-pyrazole ligand and shown that they can form stable complexes with Tb<sup>3+</sup> ion. Differences in the IR spectra of the free ligands and the metal complexes indicate that coordination of each of the ligands was occurring at the oxygen atoms of the carbonyl, enol groups, the nitrogen atoms of the pyridine ring and the pyrazole ring. The complexes each exhibited characteristic fluorescence and the fluorescence intensities of  $Tb(L_2)_3 \cdot 4H_2O$  are twice as strong as those of  $Tb_2(L_1)_3 \cdot 5H_2O$ . The complex with ligand (L<sub>2</sub>) is an very good green luminescent material that could be used as a candidate material in organic light-emitting devices (OLEDs). Based on the results, a series of bis-pyrazoles can be synthesized and screened to optimize the luminescent properties of these lanthanide ions complexes.

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