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Preparation, characterization and luminescent properties of lanthanide complexes with a new aryl amide bridging ligand

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ABSTRACT

A new aryl amide type bridging ligand 1,4-bis{[(2′-benzylaminoformyl)phenoxyl]ethoxyl}benzene (**L**) and its complexes with lanthanide ions (Ln = Pr, Nd, Sm, Eu, Gd, Tb, Dy, Er) were synthesized and characterized by elemental analysis, infrared spectra and electronic spectra. At the same time, the luminescent properties of the Sm, Eu, Tb and Dy complexes in solid state and the Tb complex in solvents were also investigated. At room temperature, these four complexes exhibited characteristic luminescence emissions of the central metal ions under UV light excitation and could be significant in the field of supramolecular photonic devices.

1. Introduction

The emission of light from lanthanide ions arises from f–f transitions, which result in emission bands with extremely narrow bandwidth and no theoretical cap on the quantum efficiency. This makes lanthanide ions very attractive for a variety of applications, such as chromophores for LEDs and as probes and labels in a variety of biological and chemical applications [1]. And the probes based on europium and terbium ions are of special interest because of the particularly suitable spectroscopic properties of these ions, such as their large stokes shift and narrow emission profiles [2,3]. Since f–f transitions are Laporte forbidden [4], free Ln^{3+} have low extinction coefficients resulting in low luminescence intensity. Therefore, it is necessary to sensitize these cations through a suitable chromophore (“antenna effect”) [5], an area of research that has been highly active in recent years. The role of the ligands is on one hand, to collect the photons provided by the light source in order to allow an energy transfer to the emitting levels of the Ln^{3+} ion and on the other to shield it against the solvent in order to avoid non-radioactive deactivation processes. It is generally accepted that the energy transfer from ligand to Ln^{3+} ion occurs from the lowest triplet state energy level T_1 of the ligand to the resonance level of Ln^{3+} [6]. And this energy transfer process is one of the most important processes having influence on the luminescent properties of the lanthanide complexes.

Recently, polynuclear lanthanide complexes, especially with luminescent properties, are attracting more and more chemists [7]. Our group is interested in the supramolecular coordination chemistry of lanthanide ions with amide type ligands that have strong coordination capability to the lanthanide ions and terminal group effects [8]. We have designed a series of polyfunctional ligands having both selective ability to coordinate more than one lanthanide ions and enhanced luminescence of lanthanide complexes by providing some of proper conjugate absorption groups suitable for energy transfer. In the present work, we designed and synthesized a new aryl amide type bridging ligand 1,4-bis{[(2′-benzylaminoformyl)phenoxyl]ethoxyl}benzene (**L**) (Scheme 1) and studied the luminescent properties of samarium, europium, terbium and dysprosium complexes with this new ligand. Under the excitation of UV light, Sm, Eu, Tb and Dy complexes all can exhibit characteristic emission of central lanthanide ions. The lowest triplet state energy level of the ligand was calculated from the phosphorescence spectrum of the Gd complex at 77 K. The result indicate that the triplet state enengy level of this ligand matches better to the resonance level of Tb(III) than other lanthanide ions.

2. Experimental

2.1. Materials

Lanthanide nitrates [9] and *N*-benzylsalicylamide [10] were prepared according to the literature methods, respectively. Other chemicals were obtained from commercial sources and used without further purification.

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Table 1
Analytical data of the complexes (calculated values in parentheses)

Complexes	Analysis (%)			
	C	H	N	Ln
$Pr_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	53.91 (53.59)	4.44 (4.39)	6.62 (6.33)	11.13 (11.10)
$Nd_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	53.77 (53.50)	4.43 (4.05)	6.60 (6.34)	11.30 (11.33)
$Sm_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	53.51 (53.23)	4.41 (4.29)	6.57 (6.33)	11.77 (11.75)
$Eu_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	53.44 (53.20)	4.41 (4.06)	6.56 (6.34)	11.86 (11.86)
$Gd_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	53.22 (52.96)	4.39 (4.03)	6.53 (6.28)	12.25 (12.22)
$Tb_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	53.15 (52.93)	4.38 (4.07)	6.52 (6.32)	12.37 (12.34)
$Dy_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	53.01 (52.87)	4.37 (4.14)	6.51 (6.40)	12.56 (12.58)
$Er_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	52.81 (52.47)	4.35 (4.58)	6.48 (6.47)	12.87 (12.90)

Table 2
The IR spectra data (cm^{-1})

Compounds	$\nu(C{=}O)$	$\nu(Ar{-}O{-}C)$	$\nu(NO_3{}^-)$				
			ν_1	ν_4	ν_2	ν_5	$\nu_1-\nu_4$
L	1632	1233, 1110					
$Pr_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	1610	1224, 1112	1485	1294	1029	816	191
$Nd_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	1610	1225, 1112	1486	1296	1029	816	190
$Sm_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	1610	1225, 1112	1484	1300	1029	815	184
$Eu_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	1610	1226, 1113	1480	1302	1030	814	178
$Gd_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	1611	1226, 1113	1482	1303	1031	815	179
$Tb_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	1609	1226, 1112	1481	1303	1031	815	178
$Dy_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	1611	1225, 1112	1482	1305	1031	814	177
$Er_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$	1612	1225, 1113	1486	1308	1032	817	178

2.2. Methods

The lanthanide ions were determined by EDTA titration using xylenol orange as an indicator. Carbon, nitrogen and hydrogen were determined on an Elementar Vario EL analyzer (see Table 1). The FT-IR spectra were recorded on a Nicolet 360 FT-IR spectrometer using KBr pellets in the range of 4000–400 cm^{-1} (see Table 2). ^1H NMR spectrum was measured on a Varian Mercury-300B spectrometer in *d*-chlorform solution with TMS as internal standard. Electronic spectra were recorded with a Varian Cary 100 spectrophotometer in methanol solution. Luminescence and phosphorescence spectra were obtained on a Hitachi F-4500 fluorescence spectrophotometer.

2.3. Synthesis of the ligand *L*

The synthetic route for the ligand **L** is shown in Scheme 1. The intermediate **A** [11] and **B** [12] were prepared according to literature methods. *N*-Benzylsalicylamide (5 mmol), potassium carbonate (5 mmol) and acetone (50 cm^3) were warmed to ca. 60 °C, then the intermediate **B** (15 mmol) was added. The reaction mixture was stirred and refluxed for 12 h. After cooling down, the mixture was filtered to give the crude product, which was washed by distilled water to afford pure ligand **L** as a white solid, yield 63%, m.p. 168–170 °C. ^1H NMR ($CDCl_3$, 300 MHz): δ: 4.16(t, J = 9.0 Hz, 4H), 4.40(t, J = 9.3 Hz, 4H), 4.57(d, J = 5.7 Hz, 4H), 6.64(s, 4H), 6.96–7.47(m, Ar–H, 16H), 8.23(d, J = 1.8 Hz, 1H), 8.26(d, J = 1.8 Hz, 1H), 8.42(b, 2H); IR (KBr pellet, cm^{-1}) ν: 3296(w, N–H), 3032(m, Ar–H), 1632(s, C=O), 1601(s), 1539(m), 1474(m), 1452(m), 1233(s, Ar–O), 1110(s, Ar–O), 760(s) and 694(m).

2.4. Preparation of the complexes

A solution of 0.15 mmol **L** in 10 cm^3 of ethyl acetate was added drop wise to a solution of 0.1 mmol lanthanide nitrate (Ln = Pr, Nd, Sm, Eu, Gd, Tb, Dy, Er) in 10 cm^3 of ethyl acetate. The mixture was stirred at room temperature for 4 h. Then diethyl ether was added to the mixture and the precipitated solid complex was filtered, washed with ethyl acetate and diethyl ether and dried in vacuo over P_4O_{10} for 48 h and submitted for elemental analysis, yield: 72%.

3. Results and discussion

3.1. Properties of the complexes

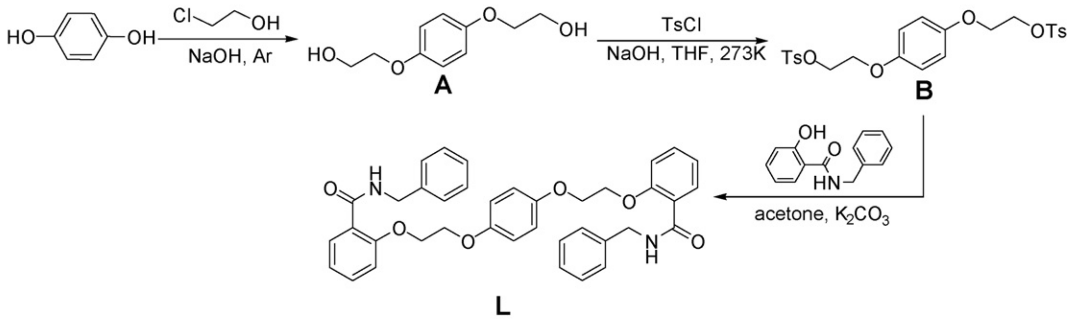
Analytical data for the newly synthesized complexes, listed in Table 1, indicate that these eight complexes of lanthanide nitrate all conform to a 2:3 metal-to-ligand stoichiometries, $Ln_2\mathbf{L}_3(NO_3)_6 \cdot 2H_2O$. All complexes are soluble in DMF, DMSO and methanol, but slightly soluble in ethanol, ethyl acetate, acetonitrile, acetone, chloroform, THF and diethyl ether.

3.2. IR spectra

The main infrared bands of the ligand and the complexes are presented in Table 2. The complexes have similar IR spectra, of which the characteristic bands have similar shifts (see Table 2), suggesting they have a similar coordination structure.

The IR spectrum of the free ligand shows bands at 1632, 1233 and 1110 cm^{-1}, which may be assigned to $\nu(C{=}O)$ and $\nu(Ar{-}O{-}C)$, respectively. In the complexes, the low-energy band remains unchanged, but the high-energy band red shifts to about 1610 cm^{-1} ($\Delta\nu = 22\ cm^{-1}$) as compared to its counterpart for the “free” ligand, indicating that only the oxygen atom of C=O takes part in coordination to the metal ions.

The characteristic frequencies of the coordinating nitrate groups (C_{2v}) appear at about 1485 cm^{-1} (ν_1), 1030 cm^{-1} (ν_2), 815 cm^{-1} (ν_3) and 1300 cm^{-1} (ν_4) and the difference between the two highest frequency bands (ν_1 and ν_4) is about 180–190 cm^{-1}, clearly establishing that the $NO_3{}^-$ groups in the solid complexes coordinate to the lanthanide ion as bidentate ligands [13]. Additionally, no bands at 1380, 820 and 720 cm^{-1} in the spectra of complexes indicates that free nitrate groups (D_{3h}) are absent.



Scheme 1. The synthetic route for the ligand **L**.

Table 3
Electronic spectral data and covalent parameters of Pr, Nd and Er complexes

Complexes	Frequency (cm^{-1})	Assignment	Covalent parameters
$Pr_2\mathbf{L}_3(NO)_6{\cdot}2H_2O$	21,277	$^3H_4 \rightarrow {}^1I_6$	$\beta = 0.9940$
	20,661	$^3H_4 \rightarrow {}^3P_1$	$\delta = 0.2506$
	16,611	$^3H_4 \rightarrow {}^1D_2$	$b^{1/2} = 0.05657$
$Nd_2\mathbf{L}_3(NO)_6{\cdot}2H_2O$	17,212	$^4I_{9/2} \rightarrow {}^2G_{7/2}$	$\beta = 0.9969$
	13,369	$^4I_{9/2} \rightarrow {}^4S_{3/2}$	$\delta = 0.3110$
	12,547	$^4I_{9/2} \rightarrow {}^2H_{9/2}$	$b^{1/2} = 0.03937$
$Er_2\mathbf{L}_3(NO)_6{\cdot}2H_2O$	26,525	$^4I_{15/2} \rightarrow {}^2G_{11/2}$	$\beta = 1.0067$
	20,534	$^4I_{15/2} \rightarrow {}^4F_{7/2}$	$\delta = -0.6631$
	19,194	$^4I_{15/2} \rightarrow {}^2H_{11/2}$	
	15,337	$^4I_{15/2} \rightarrow {}^4F_{9/2}$	

In the IR spectra of the complexes, broad bands at 3350 cm^{-1} occur from the O–H stretching vibrations of water and the absence of a band for $\rho_r(H_2O)$ at ca. 590 cm^{-1} shows that the water molecules in the complexes are not coordinated water [14].

3.3. Electronic spectra

The electronic spectra in the visible region of the Ln(III) complexes exhibit alternations in intensity and shifts in position of the absorption bands relative to the corresponding Ln(III) aquo ions. The shift has been attributed by Jørgensen to the effect on the crystal field of interelectronic repulsion between the 4*f* electrons, and is related to the covalent character of the metal–ligand bond, assessed by Sinha's parameter (δ), the nephelauxetic ration (β) and the bonding parameter ($b^{1/2}$) [15].

Absorption spectra of the Pr(III), Nd(III) and Er(III) complexes were registered in methanol solutions at room temperature and the covalent parameters were calculated and listed in Table 3. The values of β, δ and $b^{1/2}$ indicate that the interaction between the trivalent lanthanide ions and the ligands is essentially electrostatic and that there is a very small participation of 4*f* orbitals in bonding [16]. Moreover, the magnitudes of covalency and bonding parameters increase from Pr(III) to Er(III), indicating that the extent of electrostatic character of the metal–ligand bond increases with atomic number (in agreement with the lanthanide contraction).

3.4. Luminescent properties of the complexes

Excited by the absorption band at 323 nm, the "free" ligand exhibits broad emission bands ($\lambda_{max} = 442$ nm) in solid state (the excitation and emission slit widths were 2.5 nm). The luminescence emission spectra of $Ln_2\mathbf{L}_3(NO_3)_6{\cdot}2H_2O$ (Ln = Sm, Eu, Tb, Dy) in solid state (the excitation and emission slit widths were 1.0 nm, Table 4 and Fig. 1a–d) were recorded at room temperature. It is shown in Fig. 1 that these four complexes all can show the characteristic emissions of the central metal ions, indicating that the ligand **L** is a good organic chelator to absorb and transfer energy to metal ions. Intramolecular energy transfer from the triplet state of the ligand to the resonance level of the Ln(III) ion is one of the most important processes influencing the luminescence quantum yields

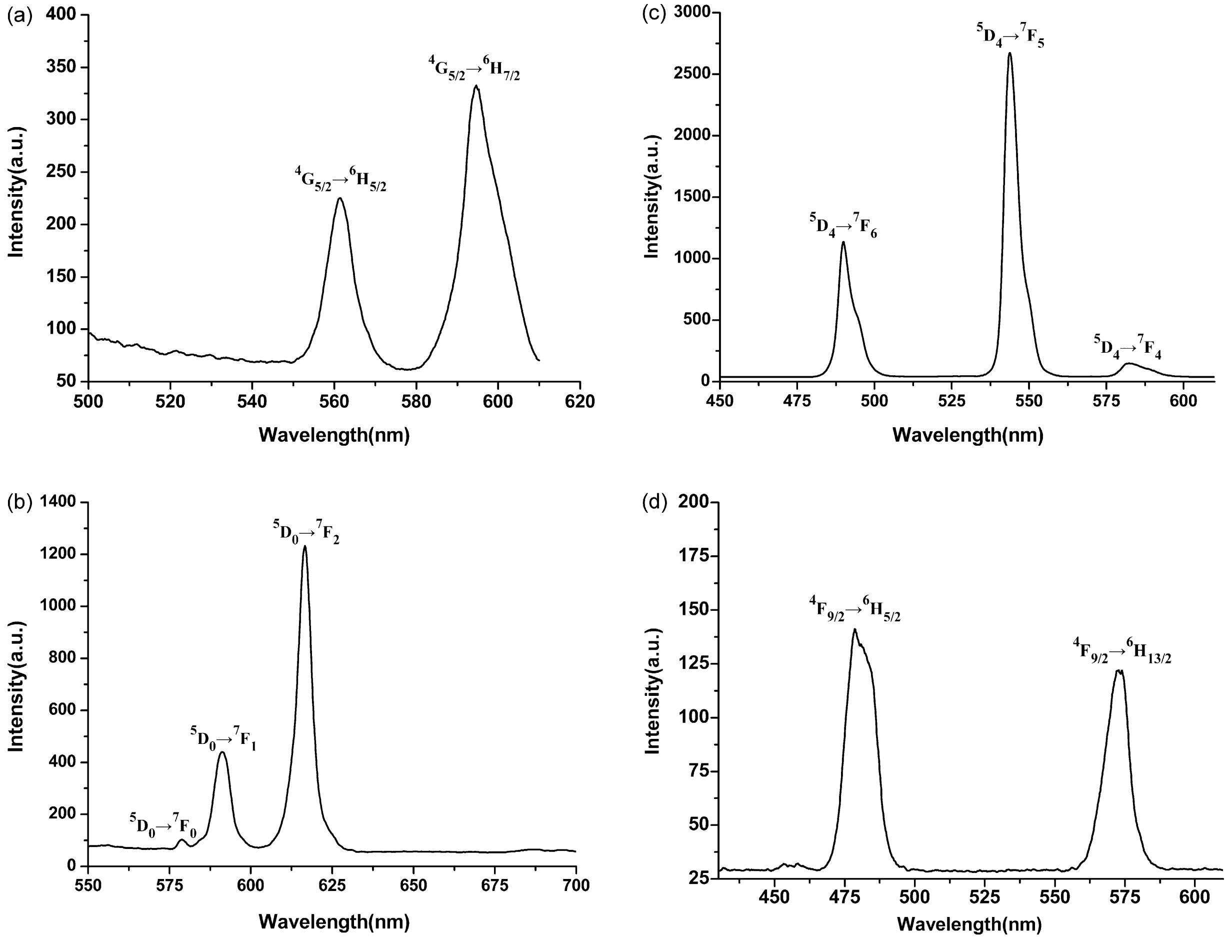


Fig. 1. Emission spectra of the complexes $Ln_2\mathbf{L}_3(NO)_6{\cdot}2H_2O$ in solid state at room temperature: (a) $Sm_2\mathbf{L}_3(NO)_6{\cdot}2H_2O$; (b) $Eu_2\mathbf{L}_3(NO)_6{\cdot}2H_2O$; (c) $Tb_2\mathbf{L}_3(NO)_6{\cdot}2H_2O$; (d) $Dy_2\mathbf{L}_3(NO)_6{\cdot}2H_2O$.

Table 4
Luminescence data for Sm, Eu, Tb and Dy complexes

Compounds	λ_{ex} (nm)	λ_{em} (nm)	RLI[a]	Assignment
L	323	442	8087	
Sm_2**L**$_3(NO)_6 \cdot 2H_2O$	320	561	230	$^4G_{5/2} \rightarrow {}^6H_{5/2}$
		594	340	$^4G_{5/2} \rightarrow {}^6H_{7/2}$
Eu_2**L**$_3(NO)_6 \cdot 2H_2O$	396	579	108	$^5D_0 \rightarrow {}^7F_0$
		591	441	$^5D_0 \rightarrow {}^7F_1$
		617	1259	$^5D_0 \rightarrow {}^7F_2$
Tb_2**L**$_3(NO)_6 \cdot 2H_2O$	323	490	1173	$^5D_4 \rightarrow {}^7F_6$
		544	2726	$^5D_4 \rightarrow {}^7F_5$
		582	153	$^5D_4 \rightarrow {}^7F_4$
Dy_2**L**$_3(NO)_6 \cdot 2H_2O$	323	479	145	$^4F_{9/2} \rightarrow {}^6H_{5/2}$
		572	128	$^4F_{9/2} \rightarrow {}^6H_{13/2}$

[a] RLI: relative luminescence intensity.

of lanthanide complexes [17]. This ligand has multiple aromatic rings with a semirigid skeleton structure, so it is a strong luminescence substance [18]. The ligand-based emission band in the Tb complex is invisible, which means the ligand-to-metal energy transfer in the Tb^{3+} complex is most efficient in these complexes. In the spectrum of Eu complex, the relative intensity of $^5D_0 \rightarrow {}^7F_2$ is more intense than that of $^5D_0 \rightarrow {}^7F_1$, showing that the Eu(III) ion lie in a non-centrosymmetric coordination site [19]. And the appearance of the $^5D_0 \rightarrow {}^7F_0$ transition is common when europium exists in a non-centrosymmetric ligand field allowing both electric- and magnetic-dipole transitions to mix resulting in transitions to the "forbidden" state [20].

A triplet excited state T_1 which is localized on one ligand only and is independent of the lanthanide nature [21]. In order to acquire the triplet excited state T_1 of the ligand **L**, the phosphorescence spectrum of the Gd^{3+} complex was measured at 77 K in a methanol–ethanol mixture (1:1, v/v). And the triplet state energy level T_1 of the ligand **L**, which was calculated from the shortest-wavelength phosphorescence band [6], is 24,331 cm^{-1}. This energy level is above the lowest excited resonance level $^4G_{5/2}$ (17,924 cm^{-1}) of Sm^{3+}, $^5D_0$ (17,286 cm^{-1}) of Eu^{3+}, $^5D_4$ of (20,545 cm^{-1}) Tb^{3+} and $^4F_{9/2}$ (21,144 cm^{-1}) of Dy^{3+}. Thus the absorbed energy could be transferred from the ligand to the Sm, Eu, Tb or Dy ions. And we may deduce that the triplet state energy level T_1 of this ligand **L** matches better to the lowest resonance level of Tb^{3+} ($\Delta\nu = 3786\,cm^{-1}$) than to Sm^{3+} ($\Delta\nu = 6407\,cm^{-1}$), Eu^{3+} ($\Delta\nu = 7045\,cm^{-1}$) and Dy^{3+} ($\Delta\nu = 3187\,cm^{-1}$) ions, because such large or small $\Delta\nu$ could result in the non-radiative deactivation of the lanthanide emitting state and quench the luminescence of the complexes [22].

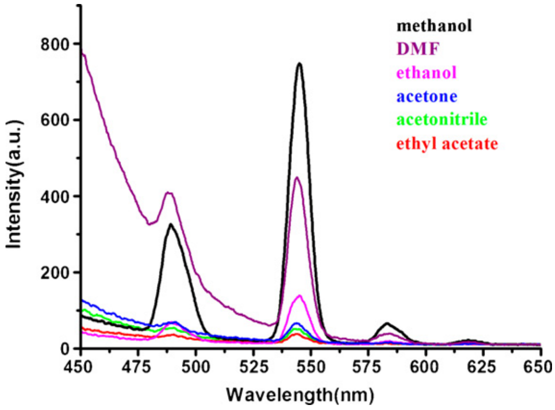


Fig. 2. Emission spectra of the Tb^{3+} complex in different solutions at room temperature.

The luminescence emission spectra of the Tb^{3+} complex in DMF, methanol, ethanol, acetonitrile, acetone and ethyl acetate solutions (concentration: 1.0×10^{-3} mol L^{-1}, the excitation and emission slit widths were 5.0 nm, Fig. 2) were recorded at room temperature. It could be seen from Fig. 2 that in methanol solution the Tb complex has the strongest luminescence, and then in DMF, ethanol, acetone, acetonitrile and ethyl acetate. This is due to the coordinating effects of solvents, namely solvate effect [23]. Together with the raising coordination abilities of methanol, DMF, ethanol, acetone, acetonitrile and ethyl acetate for the lanthanide ions, the oscillatory motions of the entering molecules consume more energy which the ligand triplet level transfer to the emitting level of the lanthanide ion.

4. Conclusions

According to the data and discussion above, the aryl amide type bridging ligand 1,4-bis{[(2′-benzylaminoformyl)phenoxyl]ethoxyl}benzene (**L**) can form stable solid complexes with lanthanide nitrates. When the ligand formed the lanthanide complexes, obvious changes in IR spectra were observed. In the complexes, lanthanide ions were coordinated to the C=O oxygen atoms of the ligand **L**. Based on the structure of the ligand and the stoichiometries of the complexes, we deduce that ligand **L** acts as a bi(unidentate) bridging ligand connecting two lanthanide(III) ions in these complexes and the coordination polymers were generated. The luminescent properties of the Sm, Eu, Tb and Dy complexes in solid state were investigated. Under the UV light excitation, the complexes exhibited characteristic luminescence of samarium, europium, terbium and dysprosium ions. This indicates that the ligand **L** is a good organic chelator to absorb and transfer energy to metal ions. The lowest triplet state energy level of the ligand indicates that the triplet state energy level (T_1) of the ligand matches better to the resonance level of Tb(III) than other lanthanide ions. Thus, the present results demonstrate that the 1,4-bis{[(2′-benzylaminoformyl)phenoxyl]ethoxyl}benzene complexes of lanthanide nitrates may find potential applications in the field of photonic devices.

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