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Synthesis, characterization, photophysical properties of lanthanide complexes with flexible tripodal carboxylate ligands

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Dedicated to Alfred Werner on the 100th Anniversary of his Nobel prize in Chemistry in 1913.

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1. Introduction

The syntheses of lanthanide(III) complexes have been under much spotlight due to their photoluminescent properties, with europium and terbium complexes playing a prominent role [1]. Luminescent materials made with lanthanide(III) complexes have been extensively used in optoelectronics [2] as well as in biological applications such as the detection of various bioactive molecules and *in vitro* imaging [3]. Emissive lanthanide(III) complexes are taking over most organic-based imaging probes due to their unique properties, e.g. long emission lifetimes, large Stokes shifts and fingerprint emission patterns. In combination with time-resolved microscopy, luminescence from lanthanide(III)-based imaging probes becomes differentiable from biological autofluorescence which leads to better imaging qualities.

Since lanthanide $4f^{N}-4f^{N}$ transitions are Laporte forbidden, the direct excitation of electrons in tripositive lanthanide ions is inefficient. In order to overcome such problem and achieve enhanced luminescence, a sensitizing chromophore, so-called an antenna, is used as a ligand to chelate to the emissive lanthanide center [4]. Hammila had reported long ago that the antenna effect could also occur by two-photon excitation of an appropriate antenna [5] and therefore it is important to design antennae that could produce efficient antenna effect. The binding strength of the ligands [6],

ABSTRACT

Three series of lanthanide complexes $[LnL_1, LnL_2 \text{ and } LnL_3 Ln = Eu, Gd and Tb]$ have been synthesized with tripodal amide ligands. These ligands have similar benzoate antenna with different substituent groups at para-position ($L_1 = -OME, L_2 = -H; L_3 = -CF_3$). The antenna effect is known to play a prominent role in the luminescence of the lanthanide ions emitting in the coordination complexes. The effect of the ligands triplet and antenna effect have been studied here. The room and low temperature photophysical properties of the nine complexes have been monitored in solution with excitation at 330 nm. The emission antenna effect (quantum yield and emission intensity) varied with the triplet state of ligand that comes from the substituent on the benzoate antenna.

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the ultraviolet (UV) absorption properties absence of high frequency vibrational modes [7], and location of donor excited states of the ligand [7] identity are important criteria to be considered [8]. Because the high phototoxicity of UV excitation is undesirable when undergoing bio-imaging [9]. The tailoring of excited state energies by substituents associated with coordinating *benzoate* ligands has yielded increased emission quantum yields [10]. However, there is a lack of systematic studies for the emission quantum yield *via* linear excitation regarding the change of the substituent groups in the same benzoate tripodal amide backbone.

Herein, we have synthesized nine lanthanide complexes with three tripodal amide ligands and three lanthanide salts respectively (Ln = Eu, Gd and Tb). Three tripodal amide ligands are conjugated with a benzoate chromophore, with the only variations at the para-substituent groups ($L_1 = OCH_3$, $L_2 = H$, $L_3 = CF_3$). The nine lanthanide complexes are designated as Ln-L1, Ln-L2, Ln-L3 to indicate complexion with different chromophores, where Ln = Eu, Gd and Tb (Fig. 1). The mass spectra of the complexes are summarized in the experimental section and supporting information. The environmental-hypertensive emission spectra of the three europium complexes have been examined in solution to investigate the coordination environment/numbers of chelates on the europium(III) ion. All the spectral measurements were conducted in DMSO, since aqueous environment will quenching the luminescence instensity significantly [11]. The photophysical properties of these lanthanide complexes have been compared via linear excitation. The triplet states of these complexes have been evaluated with low temperature emission spectra with three Gd complexes (See Table 1).



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Fig. 1. The molecular structure of lanthanide complexes $Ln-L_1$, $Ln-L_2$ and $Ln-L_3$. (Ln = Eu, Gd and Tb).

2. Results and discussion

2.1. Synthesis and characterization

A series of tripodal amide ligands are based on N-[2-(bis{2-[(benzoyl)amino]ethyl}amino)-ethyl]benzamide, with various substituent groups in the para position (Fig. 2) were obtained from tris(2-aminoethyl)amine with different species of methyl benzoate with catalysis of thionyl chloride under room temperature. L₃ gave crystals for structural analysis and are shown in Fig. 1b. ¹H NMR and ¹³C NMR suggest structural information of these ligands (Supplementary Figs. S1–S3). The product yields were found to be approximately over ~70%.

Single crystals of the ligand **L**₃, which are suitable for X-ray analysis, were obtained from slow evaporation of dichloromethane solution at room temperature for a few days. Selected bond-distances and bond angles are listed in supporting information Table S2.

Fig. 2 shows a perspective view of L_3 with the packing diagrams of L_3 projected down the c-axis. The three tripodal arms of the ligand are extended, in spite of the intramolecular hydrogen bonding that involves the amide N1-H1 group and both N4 and O3.

Table 1			
Selected bond length	(Å, left) and ang	le (right) of	ligand L

La				
0(1)	C	(3)		1 233(5)
O(2)		(13)		1 230(6)
N(1)		(2)		1 457(6)
N(1)	H	(5)		0.84(5)
N(2)	C	(13)		1.324(6)
N(3)		(22)		1.321(3) 1.449(7)
N(3)	H	(22)		0.88(5)
N(4)	C	(11)		1.466(5)
C(1)	C	(2)		1.514(6)
C(4)	C	(5)		1.381(6)
C(5)	C	(6)		1.383(8)
C(6)	C	(7)		1.370(9)
C(7)	C	(8)		1.381(8)
C(8)	C	(9)		1.381(9)
C(13)	C((14)		1.495(7)
C(14)	C((19)		1.383(6)
C(15)	H	l(15)		0.950(6)
C(16)	H	l(16)		0.950(8)
C(17)	C((20)		1.48(1)
C(21)	C((22)		1.518(8)
La				
£3 C(6)	0(5)		C(4)	122 1(3)
C(5)	O(4)		C(9)	1174(2)
C(4)	0(3)		O(1)	1194(2)
O(1)	O(3)		N(2)	1205(2)
C(2)	N(2)		C(3)	122.2(2)
C(2)	N(2)		H(6)	120(1)
C(3)	N(2)		H(6)	116(1)

Additional hydrogen bonding is depicted in Fig. 1c. The amide N3–H3 group forms a centrosymmetrically related pair of hydrogen bonds between H3 and O2'. The amide N2–H2 group hydrogen bonds to O1" in a molecule that is translated along the *a*-axis.

It is interesting to note that the tripodal molecule is quite rigid and not flexible; this is due to the extensive intramolecular hydrogen bonding. There is also an intramolecular hydrogen bond between a carboxamido N–H group of one arm of the tripodal ligand and a carbonyl O atom of a second carboxamide group [12].

2.2. Photophysical properties of lanthanide complexes

The solution state electronic absorption, emission, and excitation spectra were recorded for the lanthanide ($Ln = Eu^{3+}$, Gd^{3+} and Tb^{3+}) complexes at room temperature. The UV-absorption bands of the complexes were red-shifted ~8 nm after complexation. The extinction coefficients of all nine lanthanide complexes are listed in Table 2. The complexes with benzyl chromophore (L_2) have shown the highest extinction coefficient at 280 nm (~2.1 mmol⁻¹cm⁻¹) in their own series (i.e. europium complexes in Eu L_1 , Eu L_2 , Eu L_3 , Table 2). The excitation spectra (Fig. 3b) of the complexes presented similar bands to their absorption spectra, located at ~280 nm and 330 nm, attributed to intraligand excitations [13].

The room temperature $4f^N$ emission spectra of europium and terbium complexes under excitation at 330 nm (similar absorption extinction of all nine complexes) are readily assigned (Fig. 4). For Tb complexes, four structured narrow green emission bands at 480, 545, 580, 620 nm of Tb³⁺ (Fig. 4a) are assigned to an electronic transitions ⁵D₄ to ⁷F_J (*J* = 6, 5, 4, 3), whereas bands at 594, 620 and 700 nm of Eu³⁺ complexes correspond to ⁵D₀ to ⁷F_J (*J* = 1., 2, 3, 4) (Fig. 4b).

Rather than mass spectroscopy, analysis of the europium emission spectral profiles of three europium complexes allowed certain information of their coordination environment to be obtained. In literature, there are numbers of studies to show the sensitivity of Ln^{3+} to its coordination can be inferred from their emission spectra, even for Tb^{3+} (4f⁸, electrons are inner, shield electrons), the environment can also be inferred by monitoring their low temperature electronic spectra [14]. In europium, room temperature emission spectra is available to give more instructive coordination information due to the higher energy transitions from the non-degenerate ⁵D₀ level which is fairly simple.

In Fig. 4b, the relative intensity of the electric-dipole allowed $\Delta J = 2$ (~618 nm) transitions compared to the $\Delta J = 1$ magnetic dipole transitions was almost constant. (EuL₁ = 1:9.2; EuL₂ = 1:2.2; EuL₃ = 1:2.3) The relative intensity of the hypersensitive J = 2 transitions allow us to determine the symmetry and the polarisability of the capping axial donor. The slight variation may be induced by the steric hindrance of the three substituents groups in the ligand [15]. We also compared the relative emission intensity ratios of $\Delta J = 1$ to $\Delta J = 2$ with our previously reported polymeric europium complexes (with known molecular structures) that are formed by the same ligand L₂ and information are shown in the Fig. S4. The ratio of $\Delta J = 1$ to $\Delta J = 2$ in the polymeric form is around 1:6 [16].

The comparison of luminescence quantum efficiency of lanthanide complexes were previously based upon the energy of the respective ligand triplet state and the lanthanide emissive states [17]. In the present study, the triplet state of three antenna are affected by their substituent groups, and governed the variation of emission quantum efficiency. We have chosen excitation at 330 nm since all three chromophores showed similar absorption coefficients at that wavelength. The emission quantum yields of terbium and europium analogues are summarized in the Table 2. The emission quantum yields are obtained by a commercially available integrated sphere [18].



Fig. 2. The ORTEP diagram and hydrogen bonding of L₃.

Table 2The photophysical properties of LnL_1 , LnL_2 , LnL_3 (Ln = Eu, Gd and Tb).

	ϵ (DMSO, mmol ^{-1} cm ^{-1})	τ (DMSO, ns) ^a	τ (DMSO, 77 K μs)ª	φ (DMSO,%) ^a
Eu-L ₁	1.01	-	_	1.2
Eu-L2	1.54	-	-	3
Eu-L3	2.1	-	-	1.5
Gd-L ₁	1.03	6.7	21	-
Gd-L ₂	1.50	5.5	18	-
Gd-L ₃	2.05	5.8	16	-
Tb-L ₁	1.06	-	-	1.5
Tb- L ₂	1.48	-	-	1.5
Tb- L 3	2.1	-	-	3.3

^a With 330 nm excitation.

Terbium (TbL₃) complexes with a trifloromethyl group at the chromophore ($-CF_3$, L₃) gave the highest quantum yields among their series with excitation at 330 nm, but in europium complexes, EuL₂ is the brightest one. In this study, the triplets of these europium or terbium complexes have been determined through the low temperature emission spectra from their Gd analogues (GdL₁, GdL₂ and GdL₃) in order to examine in details the geometry and electronic structure of the ligand and the number of

chromophores coordinated. The lowest energy level of Gd^{3+} (${}^{6}P_{7/2}$) is much higher than the energy levels of the ligands; therefore, it is reasonably assumed that no energy transitions take place between the ligand and metal [19]. The phosphorescence of these Gd complexes was measured under cold conditions (77 K) in solutionmatrix state. Strong emission bands of the three Gd complexes are located at 395 nm, 383 nm and 380 nm for GdL₁–L₃, which were blue-shifted for ~40 nm compared with their room temperature emission spectra (Fig. 5). The luminescence lifetimes were recorded to verify their fluorescent or phosphorescent nature. The emission lifetimes measured for ca. 405 nm were on a nanosecond scale ($S_1 \rightarrow S_0$) whereas that at ca. 450 nm were on a microsecond scale ($T_1 \rightarrow S_0$) for the gadolinium complexes GdL₁, GdL₂ and GdL₃.

In all three terbium and europium complexes, the energy gap between the triplet states of the chromophore/antenna to Tb (~20500 cm⁻¹) and Eu (17500 cm⁻¹) are greater than 2000 cm⁻¹, hence no back energy transfer occurred. However, the energy level of the ⁵D₀ state for Eu³⁺ is ca. 17500 cm⁻¹, which is 3000 cm⁻¹ lower than the Tb³⁺ (⁵D₄) energy level so the energy gap between T₁ and ⁵D₀ in EuL₃ (-CF₃) becomes ~6000 cm⁻¹ and compared to that between T₁ and ⁵D₄ in TbL₃ (-CF₃) which is ~2500 cm⁻¹, it is reasonable that the energy transfer from the ligand is better converted *via* the ligand triplet [20] and thus Tb complex TbL₃ exhibit better luminescence properties.



Fig. 3. (a) The UV absorption spectra and (b) excitation spectra of complexes EuL₁, EuL₂, EuL₃ in a solution of DMSO (0.1 mM).



Fig. 4. The emission spectra of (a) terbium complexes TbL₁, TbL₂, TbL₃ and (b) europium complexes EuL₁, EuL₂, EuL₃ in a solution of DMSO with excitation at 330 nm. (0.5 mM, abs_{330nm} = 0.1).

3. Conclusion

In conclusion, nine lanthanide complexes with tripodal amide ligands have been synthesized and the crystal structure of one tripodal amide ligand L_3 was obtained. The nine complexes differ in their substituents groups on the benzoate antenna which is shown to influence the electronic density of the ring and hence the luminescent properties.

4. Experimental

4.1. Synthesis of Tris[2-(benzoylamino)ethyl]amine (L1)

Benzoyl chloride (0.32 ml, 2.74 mmol) was dissolved in dry DCM (5 ml), then dropped into the solution of N1,N1-bis(2-amino-

ethyl)ethane-1,2-diamine (100 mg, 0.68 mmol) and triethylamine (1 ml, 6.8 mmol) in dry DCM (10 ml) at 0 °C under nitrogen gas. The resulting solution was stirred more 5 h at rt. and washed with 0.5 M NaOH aq. and 0.5 M HCl. The organic layer was dried over anhydrous sodium sulfate, filtered and concentrated. Silica gel flash column chromatography (DCM: MeOH = 20:1 of the residue gave a pale yellow solid (284 mg, 0.62 mmol, 91%) as the title product. ¹H NMR (CDCl₃, 400 MHz): δ 7.65 (dd, *J* = 4, 8 Hz, 6H), 7.30 (m, 3H), 7.19 (s, 3H), 7.08 (m, 6H), 3.57 (q, *J* = 6 Hz, 6H), 2.74 (t, *J* = 8 Hz, 6H); ¹³C NMR (CDCl₃, 400 MHz): δ 168.2, 133.7, 131.1, 128.2, 127.0, 53.7, 37.9. ESI-MS = 458.2.

4.2. Synthesis of Tris[2-(4-meoxyl-benzoylamino)ethyl]amine (L_2)

4-methoxybenzoyl chloride (0.32 ml, 2.74 mmol) was dissolved in dry DCM (5 ml), then dropped into the solution of



Fig. 5. The emission spectra of gadolinium complexes (GdL1-GdL3) in a solution of Me-THF with excitation at 330 nm at 298 K and 77 K. (0.5 mM).

N1,N1-bis(2-aminoethyl)ethane-1,2-diamine (100 mg, 0.68 mmol) and triethylamine (1 ml, 6.8 mmol) in dry DCM (10 ml) at 0 $^{\circ}$ C under nitrogen gas. The resulting solution was stirred more 5 h at rt. and washed with 0.5 M NaOH aq. and 0.5 M HCl. The organic layer was dried over anhydrous sodium sulfate, filtered and concentrated. Silica gel flash column chromatography.

L₂ gave a white solid (335 mg, 0.61 mmol, 90%) as the title product. ¹H NMR (CDCl₃, 400 MHz): δ 7.57 (d, *J* = 4, Hz, 6H), 7.04 (t, *J* = 6 Hz, 3H), 6.51 (d, *J* = 4 Hz, 6H), 3.72 (s, 9H), 3.57 (q, *J* = 6 Hz, 6H), 2.73 (t, *J* = 6 Hz, 6H); ¹³C NMR (CDCl₃, 400 MHz): δ 167.7, 161.7, 128.9, 126.0, 113.2, 55.1, 53.2, 37.5. ESI-MS = 548.3.

4.3. Synthesis of Tris[2-(4-trifluoromethyl-benzoylamino)ethyl]amine (L_3)

4-(Trifluoromethyl)benzoyl chloride (0.32 ml, 2.74 mmol) was dissolved in dry DCM (5 ml), then dropped into the solution of N1,N1-bis(2-aminoethyl)ethane-1,2-diamine (100 mg, 0.68 mmol) and triethylamine (1 ml,6.8 mmol) in dry DCM (10 ml) at 0 °C under nitrogen gas. The resulting solution was stirred more 5 h at rt. and washed with 0.5 M NaOH aq. and 0.5 M HCl. The organic layer was dried over anhydrous sodium sulfate, filtered and concentrated. Silica gel flash column chromatography.

L₃ gave a white solid (368 mg, 0.59 mmol, 87%) as the title product. ¹H NMR (CDCl₃, 400 MHz): δ 8.56 (t, *J* = 4 Hz, 3H), 7.87 (d, *J* = 4 Hz, 6H), 7.60 (d, *J* = 4 Hz, 6H), 3.38 (q, *J* = 8 Hz, 6H), 2.71 (t, *J* = 8 Hz, 6H); ¹³C NMR (CDCl₃, 400 MHz): δ 165.1, 138.0, 130.7, 129.4, 127.9, 125.0, 52.7, 37.5. The crystal data of **L**₃ is listed in Table S1. ESI-MS = 662.2.

4.4. Synthesis of complexes Eu-L₁, Tb-L₁ and Gd-L₁

 L_1 (20 mg, 0.044 mmol) and $Ln(NO_3)_3$ (0.044 mmol) were stirred in MeOH (2 ml) at r.t. for 24 h. Solvent was removed under vacuum, three white solid as the title complexes. Eu- L_1 = 825; Tb- L_1 = 831; Gd- L_1 = 829 [M–NO₃⁻¹].

4.5. Synthesis of complexes Eu-L₂, Tb-L₂ and Gd-L₂

 L_2 (20 mg, 0.036 mmol) and $Ln(NO_3)_3$ (0.036 mmol) were stirred in MeOH (5 ml) at rt. for 24 h. Solvent was removed under

vacuum, three white solid as the title complexes. Eu- L_2 = 737; Tb- L_2 = 743; Gd- L_3 = 828 [M-NO₃⁻¹].

4.6. Synthesis of complexes Eu-L₃, Tb-L₃ and Gd-L₃

L₃ (20 mg, 0.030 mmol) and Ln(NO₃)₃ (mmol) were stirred in MeOH (10 ml) at 50 °C for 24 h. Solvent was removed under vacuum, three white solid as the title complexes. Eu-L₃ = 837; Tb-L₃ = 843; Gd-L₃ = 841 [M-NO₃⁻¹].

4.7. Photophysical measurements

UV–Vis absorption spectra in the spectral range 200–1100 nm were recorded by a HP UV-8453 spectrophotometer. Single-photon luminescence and lifetime spectra were recorded using a Edinburgh Instruments FLS920 Combined Fluorescence Lifetime and Steady State Spectrophotometer (185–850 nm) equipped with a single photon counting photomultiplier in a Peltier Cooled Housing. The spectra were corrected for detector response and stray background light phosphorescence. The solution state quantum yields of the lanthanide complexes were measured by a demountable 142 mm (inner) diameter barium sulfide coated integrating sphere supplied with two access ports.

Low-temperature (77 K) phosphorescence spectra were obtained by exciting samples with a xenon lamp. The transparent glassy materials were formed by mixing samples with 2-methyltetrahydrofuran. The samples were placed in a tailor-made quartz tube housed in an Oxford Instruments liquid nitrogen cryostat.

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Appendix A. Supplementary data

CCDC 890169 contains the supplementary crystallographic data for L₃. These data can be obtained free of charge via http:// www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.poly.2012.07.020.

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