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Rare earth complexes with a novel ligand *N*-(naphthalen-2-yl)-*N*-phenyl-2-(quinolin-8-yloxy)acetamide: Preparation and spectroscopic studies

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ABSTRACT

Six complexes of rare earth nitrates (Ln = La, Sm, Eu, Gd, Tb, Dy) with a new amide type ligand, *N*-(naphthalen-2-yl)-*N*-phenyl-2-(quinolin-8-yloxy)acetamide (L) have been prepared and characterized by elemental analysis, conductivity measurements, IR and and ^{1}H NMR spectra. Under excitation, Eu(III) and Sm(III) complexes exhibited strong red emissions. And the luminescence intensity of Sm(III) complex is higher than that of Eu(III) complex. Thus the Eu(III) and Sm(III) complexes are the potential light conversion agent. However, the Tb(III) and Dy(III) complexes cannot exhibit characteristic emissions of terbium and dysprosium ions, respectively. The results of phosphorescence spectrum show that the triplet-state energy level of the ligand matches better to the resonance level of Eu(III) than Tb(III) ion. In addition, the luminescence of the Eu(III) complex is also relatively strong in highly diluted tetrahydrofuran solution (2×10^{-4} mol/L) compared with the powder. This is not only due to the solvate effects but also to the changes of the structure of the Eu(III) complex after being dissolved into the solvents. Furthermore, owing to the co-luminescence effect, the proper La(III) or Gd(III) doped Eu(III) complexes show stronger luminescence than the pure Eu(III) complex.

1. Introduction

Owing to their good luminescent properties, such as the large stokes shifts, narrow emission profiles, long lifetimes and so on [1], the rare earth complexes are attracting more and more attention and have been widely used in many aspects, such as light-emitting diode (LED), laser materials, optical signal amplification, fluoroimmunoassay, and so on [2–4]. Among them, the application of Eu(III) and Sm(III) complexes to light conversion agent has been paid more and more attention. It is well known that purple and green lights in the sunlight do harm to plants. The Eu(III) and Sm(III) complexes could absorb the ultraviolet light and then transform them into the beneficial red light for photosynthesis efficiently. Furthermore, the red light exhibited by Sm(III) complex fits the photosynthesis better, because of the emission peak of Sm(III) complex at about 645 nm [5,6].

However, direct excitation of Eu(III) or Sm(III) ion is not efficient because of its inherently small absorption cross-section. To overcome this problem, the ligand containing the common donor groups has been synthesized, which serves as an antenna or sensitizer, absorbing the excitation light and transferring the energy from its lowest triplet-state energy level (T) to the resonance level of Ln(III) ion [7,8]. Among numerous ligands, the amide type ligands, which are flexible in structure and have 'terminal-group effects' [9–11], will shield the encapsulated lanthanide ion from interaction with the surroundings effectively, and thus to achieve strong luminescent emissions. Therefore, an amide type ligand was selected as 'antenna' in this work. The coordination and luminescence properties of rare earth ions with the ligand have been studied in detail.

2. Experimental

2.1. Materials

The rare earth (III) nitrates were prepared from their oxide acquired from Yue Long (PR China). All other chemicals used were of analytical grade. Absolute chloroform and *N,N′*-dimethylformamide (DMF) were obtained after being distilled by standard methods.

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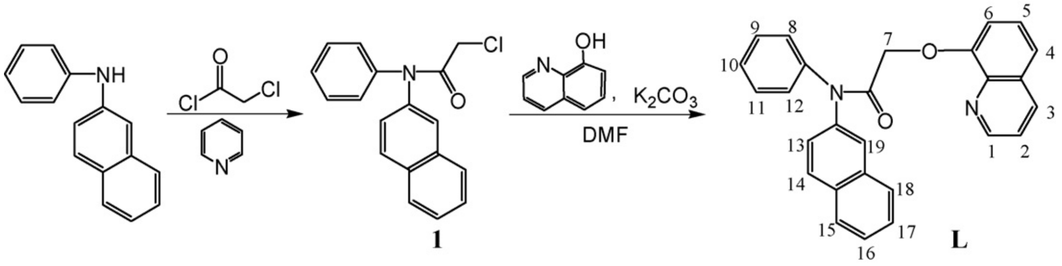

Fig. 1. Synthesis scheme of the ligand.

2.2. Physical measurements

The melting point of the ligand was determined on an XT4-100x microscopic melting point apparatus (made in Beijing, China). Elemental analyses were carried out on an Elemental Vario EL analyzer. The infrared spectra (IR, $\nu = 4000–400\,cm^{-1}$) were determined by the KBr pressed disc method on a Nicolet-170SX FT-IR spectrophotometer. ^1H NMR spectra were recorded on Bruker DRX-200 spectrometer in CD_3COCD_3 solutions. All conductivity measurements were carried out with a DDS-11A conductometer (made in China) bridge using 1.0×10^{-3} mol/L solutions in acetonitrile at 25 °C. The mass spectrum was obtained on a TRACE DSQ GC/MS. The ultraviolet absorption spectra were measured on Shimadzu UV-240 spectrophotometer. Luminescence emission and excitation spectra were determined on a Hitachi F-4500 FL spectrophotometer. Phosphorescence spectra at 77 K were taken on the same spectrophotometer equipped with phosphorescence measurement apparatus. All the complexes were dried at 100 °C to constant weights before these measurements.

3. Synthesis of ligand

The synthesis scheme is shown in Fig. 1. The compound **1** was prepared according to the literature [12].

8-Hydroxyquinoline (1.5 g, 10.3 mmol) and anhydrous potassium carbonate (1.6 g, 11.6 mmol) were added to DMF (15 mL), then **1** (2.96 g, 10.0 mmol) and a small quantity of KI were added. The reaction mixture was stirred for 5 h at 100–110 °C. After cooling down, 150 mL water was added and stirred for 2 h. The precipitate was collected by filtration and washed with water. Recrystallization from ethyl acetate gave the ligand L; Yield: 56.0%. m.p. 84–85 °C; MS, m/z: 404 $[M]^+$.

The ^1H NMR chemical shifts δ (ppm/TMS) for the protons of L in CD_3COCD_3 are listed in Table 3.

3.1. Synthesis of the complexes

The ligand (L) (0.1 mmol) and $Ln(NO_3)_3 \cdot nH_2O$ (0.05 mmol) were added to ethyl acetate (5 mL). After stirring for 4 h at room temperature, the precipitate was separated from the solution by suction filtration, purified by washing for several times with ethyl acetate, and dried for 24 h in a vacuum.

Table 1
Analytical and molar conductance data for the complexes

Complexes	C% (calc.)	H% (calc.)	N% (calc.)	Λ_m ($s\,cm^2\,mol^{-1}$)
$La_2(L)_3(NO_3)_6$	51.93(52.21)	3.32(3.25)	8.60(9.02)	227
$Sm_2(L)_3(NO_3)_6$	51.91(51.58)	3.44(3.21)	8.46(8.91)	258
$Eu_2(L)_3(NO_3)_6$	51.59(51.49)	3.56(3.20)	8.62(8.90)	270
$Gd_2(L)_3(NO_3)_6$	51.81(51.21)	3.32(3.18)	8.38(8.85)	275
$Tb_2(L)_3(NO_3)_6$	51.58(51.12)	3.34(3.18)	8.35(8.83)	278
$Dy_2(L)_3(NO_3)_6$	51.35(50.92)	3.02(3.17)	8.33(8.80)	300

4. Result and discussion

Analytical data for the complexes, presented in Table 1, conform to $Ln_2(L)_3(NO_3)_6$. All complexes are white powders, which are soluble in DMF, DMSO, acetone, methanol, acetonitrile and ethanol, slightly soluble in ethyl acetate, while hardly soluble in water and ether. The molar conductivity values of the complexes in acetonitrile are in the range of 227–300 $s\,cm^2\,mol^{-1}$, indicating that the complexes are 2:1 electrolytes [13].

4.1. IR spectra

The complexes have similar IR spectra which indicate that they have similar coordination structures (Table 2). The characteristic absorption bands of L due to ν(C=O), ν(C=N) and ν(Ar—O—C) appear at 1684, 1620 and 1260 cm^{-1}, respectively, after the formation of the complexes which shift by *ca.* 37, 29 and 2 cm^{-1}, respectively. The results clearly show that the carbonyl oxygen atom and quinoline nitrogen in the ligand L participate in the coordination to the metal ions [14,15]. However, the slight shift of ν(Ar—O—C) between each complex and the ligand indicates that the etheric oxygen atom does not coordinate with the metal ion. It is possibly due to the fact that the ligand has large sterically hindered effect, which prevents the etheric oxygen atom from coordinating with lanthanide ion.

The characteristic frequencies of the coordinating nitrate groups (C_{2v}) appear at about 1507 cm^{-1} (ν_1), 1032 cm^{-1} (ν_2), 821 cm^{-1} (ν_3), and 1312 cm^{-1} (ν_4) and the difference between the two highest

Table 2
Major IR data of the free ligand and its complexes (cm^{-1})

Compounds	ν(C=O)	ν(C=N)	ν(Ar—O—C)	$\nu_0(NO_3)$	$\nu_1(NO_3)$	$\nu_4(NO_3)$	$\nu_1–\nu_4(NO_3)$
L	1684	1620	1260				
$La_2(L)_3(NO_3)_6$	1647	1591	1260	1384	1507	1312	195
$Sm_2(L)_3(NO_3)_6$	1648	1592	1261	1382	1503	1308	195
$Eu_2(L)_3(NO_3)_6$	1642	1592	1260	1384	1503	1313	190
$Gd_2(L)_3(NO_3)_6$	1638	1592	1262	1384	1506	1312	194
$Tb_2(L)_3(NO_3)_6$	1637	1591	1262	1383	1506	1312	194
$Dy_2(L)_3(NO_3)_6$	1638	1591	1263	1384	1507	1313	194

Table 3
The ^{1}H NMR data of free ligand and its La(III) complex in CD_3COCD_3(ppm)

Compounds	H_1	H_3	H_6	H_7	$H_{2,4,5,8-19}$
L	8.85–8.88	8.25–8.30	7.17–7.21	5.03	7.43–7.98
$La_2(L)_3(NO_3)_6$	8.94–8.96	8.18–8.34	7.11–7.15	5.28	7.47–8.01

frequency bands (ν_1–ν_4) is in the range of 190–195 cm^{-1}, indicating that the coordinated nitrate groups in the complexes are bidentate [16,17]. The free nitrate groups (D_{3h}) appear at *ca.* 1384 cm^{-1} in the spectra of the complexes [18], which is in agreement with the result of the conductivity experiment.

4.2. ^{1}H NMR spectra

The ^{1}H NMR spectra of the free ligand and its La(III) complex were measured in CD_3COCD_3 at room temperature (Table 3). For L, the signals at 8.85–8.88, 7.17–7.21 and 5.03 ppm, are assigned to H_1, H_6 and H_7, respectively. Upon coordination, they are shifted by 0.08–0.09, −0.06 and 0.25 ppm, respectively. Among them, the signal of proton H_6 moves to a higher field. The abnormal phenomenon has been reported before [19], this is probably due to the reduction of the conjugation quinoline ring and tortility in the conformation of the ligand in the complex [20]. Combining with the assumptions made on the basis of the IR spectra studies, it can be concluded that the oxygen atom of the carbonyl group and quinoline nitrogen atom take part in coordination in the complexes [20,21].

According to the data of the elemental analyses, molar conductivity, IR spectra and ^{1}H NMR spectra, composition of the complexes can be inferred: $[Ln_2(L)_3(NO_3)_4](NO_3)_2$.

4.3. Luminescence properties of the complexes

4.3.1. Solids

The Eu(III) and Sm(III) complexes show strong red luminescence in solid state (Fig. 2 (solid) and Table 4). However, the Tb(III) and Dy(III) complexes only exhibit the free ligand band emission. This indicates that the ligand L is a comparative good organic chelator to absorb and transfer energy to Eu(III) and Sm(III) ions. Furthermore, it is interesting that the luminescence of Sm(III) complex is stronger than that of Eu(III) complex. Thus the Eu(III) and Sm(III) complexes are the potential light conversion agent. The high ratio value for $\eta_{Eu} = {}^5D_0 \to {}^7F_2/{}^5D_0 \to {}^7F_1$ is 2.45, showing that the Eu(III) ion does not lie in a centro-symmetric coordination site [22].

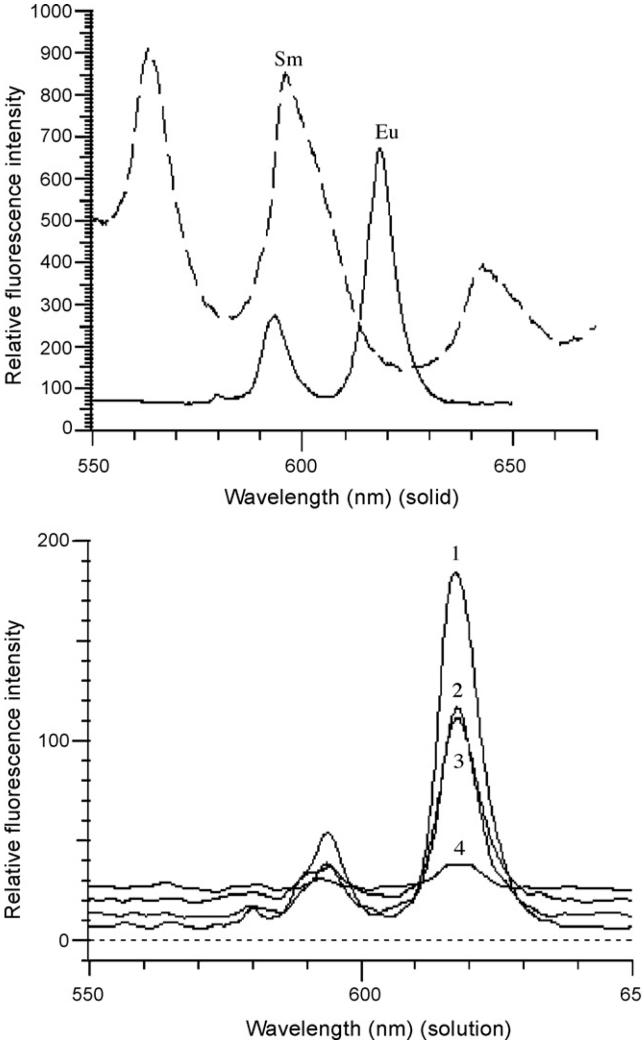

Fig. 2. (Solid) The luminescence emission spectra of Eu(III) and Sm(III) complexes in solid state at room temperature: $\lambda_{Ex(Eu)} = 337$ nm; $\lambda_{Ex(Sm)} = 343$ nm; slit width = 1 nm. (Solution) The emission spectra of Eu(III) complex in different solutions (2×10^{-4} mol/L) at room temperature: (1) tetrahydrofuran; (2) acetone; (3) acetonitrile; (4) ethanol: $\lambda_{Ex} = 337$ nm; slit width = 1 nm.

Table 4
Luminescence spectra data (nm) of the Eu(III) and Sm(III) complexes at room temperature

Compounds	Ex slit	Em slit	λ_{Ex}	λ_{Em}	Emissin intensity	Transition	Quantum yield (Φ)
$Sm_2(L)_3(NO_3)_6$[a]	1	1	343	563	909	$^4G_{5/2} \to {}^6H_{5/2}$	0.063
				596	852	$^4G_{5/2} \to {}^6H_{7/2}$	
				643	394	$^4G_{5/2} \to {}^6H_{9/2}$	
$Eu_2(L)_3(NO_3)_6$[a]	1	1	337	580	86	$^5D_0 \to {}^7F_0$	0.027
				593	275	$^5D_0 \to {}^7F_1$	
				619	673	$^5D_0 \to {}^7F_2$	
1[b]	1	1	337	617	186	$^5D_0 \to {}^7F_2$	
2[b]	1	1	337	617	118	$^5D_0 \to {}^7F_2$	
3[b]	1	1	337	618	112	$^5D_0 \to {}^7F_2$	
4[b]	1	1	337	619	40	$^5D_0 \to {}^7F_2$	
5[c]	5	5	337	618	380	$^5D_0 \to {}^7F_2$	0.027
6[c]	5	5	337	618	1428	$^5D_0 \to {}^7F_2$	0.039
7[c]	5	5	337	617	2408	$^5D_0 \to {}^7F_2$	0.081
8[c]	5	5	337	617	2506	$^5D_0 \to {}^7F_2$	0.043

[a] In solid state.

[b] In different solutions (1, tetrahydrofuran; 2, acetone; 3, acetonitrile; 4, ethanol), concentration: 2×10^{-4} mol/L.

[c] La(III) or Gd(III) doped Eu(III) complexes in tetrahydrofuran (5, the pure Eu(III) complex; 6, Eu^{3+}:La^{3+} = 0.5:0.5; 7, Eu^{3+}:Gd^{3+} = 0.9:0.1; 8, Eu^{3+}:Gd^{3+} = 0.7:0.3). The concentrations of both L and the total lanthanide ions were 1×10^{-5} mol/L.

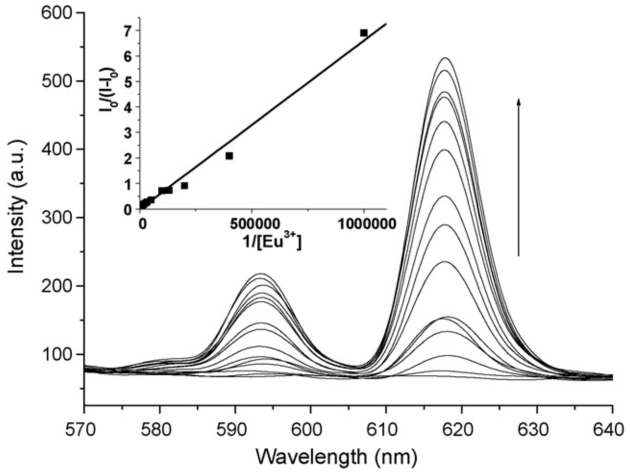

Fig. 3. Luminescence spectra of L in tetrahydrofuran (1.0×10^{-5} mol/L) in the presence of Eu^{3+} ion (as nitrate): $\lambda_{EX} = 337$ nm. The insert shows the plot of $I_0/(I-I_0)$ vs. $[Eu^{3+}]^{-1}$.

4.3.2. *Solutions*

The influences of solvents on the luminescence intensity of the Eu(III) complex were investigated. From Fig. 2 (solution) and Table 4, it could be seen that in tetrahydrofuran solution the Eu(III) complex has the strongest fluorescence, and following by that in acetone, acetonitrile and ethanol. This is due to the coordinating effects of solvents, namely solvate effect [23]. Additionally, it is interesting that the luminescence of the Eu(III) complex is also relatively strong in highly diluted tetrahydrofuran solution (2×10^{-4} mol/L). The unusual phenomenon [24,25] may be not only owing to the solvate effects but also to the changes of the structure in the Eu(III) complex after adding the solvent.

4.3.3. *Stoichiometry of ligand with Eu(III) in solution*

Fig. 3 shows the changes in the emission spectrum of L as a function of Eu^{3+} concentration in tetrahydrofuran. The insert shows the plot of $I_0/(I-I_0)$ versus $[Eu^{3+}]^{-1}$, where I_0 and I represent the emission intensity at 618 nm in the absence and presence of Eu^{3+}, respectively. The yielded straight line suggests that the complexation of Eu^{3+} into the ligand is in a 1:1 ratio and the linearity correlation coefficient R is 0.992 [26]. It clearly confirms that the structure of the Eu(III) complex in the solution is different from that in solid state (Eu^{3+}:L = 2:3). Complexation constant ($\log K$), determined from such emission spectra data for the cation, is 3.75 ± 0.05 [27,28].

4.3.4. *The triplet-state energy*

Phosphorescence spectrum of the Gd(III) complex was measured at 77 K in the methanol–ethanol (1:1) solution (1×10^{-5} mol/L). The lowest triplet-state level energy (T) of the ligand was calculated by the shortest wavelength transition in the phosphorescence spectrum to be 21,834 cm^{-1} (458 nm) [29]. This energy level is above the resonance level $^5D_1$ of Eu(III) and $^5D_4$ of Tb(III). Thus the absorbed energy could be transferred from L to the Eu(III) or Tb(III) ion. However, the intramolecular transfer efficiency depends chiefly on two energy transfer processes: one is from the lowest triplet level (T) of ligand to the resonance level of Eu(III) ion ($^5D_1$) by resonant exchange interaction, and the other is just an inverse energy transfer by the thermal de-excitation mechanism [30]. Established on this theory, the conclusion can be drawn that energy gap (ΔE) between T and ^5D (Table 5) is of opposite influence on the two energy transfer processes and an optimal value can be assumed to exist. In addition, it has been reported that [30], when $\Delta E(T-{}^5D_1)$ for Eu(III) is greater than 4000 cm^{-1} or $\Delta E(T-{}^5D_4)$ for Tb(III) is less than 1500 cm^{-1}, the fluorescence quantum yield of the Eu(III) or Tb(III) complexes is approximately zero at room temperature. According to this idea, the strong fluorescence of the Eu(III) complex is due to the optimal $\Delta E(T-{}^5D_1)$, and it should be due to the small $\Delta E(T-{}^5D_4)$ that the Tb(III) complex cannot exhibit its characteristic luminescence.

Table 5
Data of triplet-state energy and ΔE $(T-{}^5D)$

Complexes	$Gd_2(L)_3(NO_3)_6$
0–0 transition (nm)	458
Triplet-state energy level (cm^{-1})	21,834
$\Delta E(T-{}^5D_1{}^a)$ (cm^{-1})	2814
$\Delta E(T-{}^5D_4{}^b)$ (cm^{-1})	1334

[a] $^5D_1$: 19,020 cm^{-1} (the resonance level of Eu(III) ion) [36].

[b] $^5D_4$: 20,500 cm^{-1} (the resonance level of Tb(III) ion) [24].

4.3.5. *Doped complexes*

The luminescence properties of La(III) or Gd(III) doped Eu(III) complexes were measured in detail (Fig. 4 and Table 4). The concentrations of both L and the total lanthanide ions were 1×10^{-5} mol/L in tetrahydrofuran. Changing the ratio of Eu(III) to La(III) [or Gd(III)] (0.9:0.1, 0.7:0.3, 0.5:0.5, 0.3:0.7 and 0.1:0.9), the luminescence emission spectra of the ten doped complexes were measured after 12 h. It can be found that when the ratios are 0.5:0.5 (Eu^{3+}:La^{3+}), 0.9:0.1 (Eu^{3+}:Gd^{3+}) and 0.7:0.3 (Eu^{3+}:Gd^{3+}), the emission intensities of the doped complexes at 618 nm is enhanced by 3.76, 6.34 and 6.59 times compared with that of the pure Eu(III) complex, respectively. The other doped complexes only exhibit the free ligand band emission or have no notable intensity changes. This is probably due to the proper amount of La(III) or Gd(III) ion acting as an intermediate, which absorbs the triplet-state level energy of ligand and then transfers such energy to the Eu(III) ion. Such process optimizes the luminescence property of Eu ion, and is named co-luminescence effect. In addition, because the radius of Gd(III) is smaller than La(III), the distance between Gd(III) and Eu(III) ions is shorter than that between La(III) and Eu(III) ions, which results in the Gd(III) ion represents more excellent energy transfer efficiency and sensitization capability to Eu(III) [31,32].

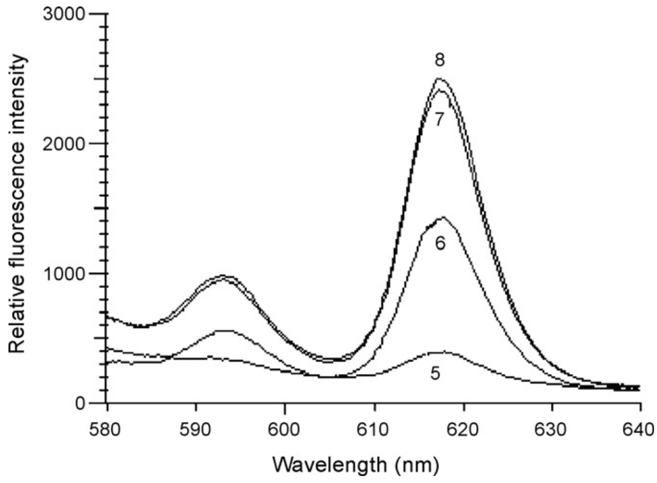

Fig. 4. Luminescence spectra of La(III) or Gd(III) doped Eu(III) complexes in tetrahydrofuran (**5**: the pure Eu(III) complex; **6**: Eu^{3+}:La^{3+} = 0.5:0.5; **7**: Eu^{3+}:Gd^{3+} = 0.9:0.1; **8**: Eu^{3+}:Gd^{3+} = 0.7:0.3). The concentrations of both L and the total lanthanide ions were 1×10^{-5} mol/L: $\lambda_{Ex} = 337$ nm; slit width = 5 nm.

4.3.6. Luminescence quantum yields

The luminescence quantum yields of the Sm(III), Eu(III) and doped complexes were determined by comparison with a calibration standard of $[Ru(bpy)_3]Cl_2$ in degassed acetonitrile solution presenting a fluorescence quantum yield of 0.062 (Φ_s) [33]. The Sm(III) complex was dissolved in acetonitrile, and the other samples were dissolved in tetrahydrofuran. The concentration of all solutions is 1×10^{-5} mol/L. Each solution was excited at λ_{max} (Table 4) on a Hitachi F-4500 FL spectrophotometer. The equation used for calculating the quantum yield, Φ_c, of the sample was [34]:

$$\Phi_c = \frac{F_c \Phi_c A_s}{F_s A_c}$$

where F denotes the integrated area under the emission spectrum; A is the absorbance at the exciting wavelength. A refractive index correction was used: $R_f{}^2$(tetrahydrofuran)/$R_f{}^2$ (acetonitrile) [35]. The values of Φ_c are listed in Table 4.

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