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# Synthesis, Characterization and Fluorescence Properties of $Y^{3+}$ -Doped Terbium Complexes with Phenoxyacetic Acid and 1,10-Phenanthroline

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**Abstract:** A series of  $Y^{3+}$ -doped terbium complexes with phenoxyacetic acid and 1, 10-phenanthroline were synthesized in anhydrous alcohol. Elemental analysis and rare earth complexometric titration showed that the composition of the complexes are  $Tb_xY_{1-x}(POA)_3phen \cdot 1/2H_2O$  ( $POA = C_6H_5OCH_2COO^-$ ,  $x = 1.0, 0.9, 0.8, 0.7, 0.6, 0.5, 0.4, 0.3, 0.2$  and  $0.1$ ). The IR absorption spectra indicated that the rare earth ions are bounded with the oxygen atoms of phenoxyacetic acid and phenoxy, and with two nitrogen atoms of 1,10-phenanthroline. Thermogram and thermogravimetric analysis indicated that the complexes release coordinated water at about 274 °C and they are stable below 576 °C. Fluorescent spectra showed that the fluorescence intensity of the series  $Tb^{3+}$  complexes can be greatly enhanced if some of terbium ions in the complexes are substituted by  $Y^{3+}$ . The fluorescence lifetime of the complexes exhibits the similar feature to the fluorescence intensities.

**Key words:**  $Y^{3+}$ -doped terbium complex; phenoxyacetic acid; 1,10-phenanthroline; fluorescence intensity

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## 1 Introduction

The luminescence of the complexes of the organic ligands with rare earth ions has become an interesting subject in the recent years<sup>[1~7]</sup>. A number of lanthanide ions display characteristic luminescence, a consequence of the f-f transitions, in the visible or near-infrared spectral regions upon ultraviolet excitation. Especially, the organic rare earth complexes, containing aroma rings as a kind of ligand, whose light absorption and energy transfer are efficient, are capable of giving of strong fluorescence<sup>[8~10]</sup>. In addition to the organic ligands, the doped rare earth complexes, which are synthesized by adding some inert cheapness rare earth ions into them, have some advantages, such as low cost and

good fluorescent property<sup>[11,12]</sup>. So far, no report was available on the synthesis and luminescence research of the complexes formed by phenoxyacetic acid and 1, 10-phenanthroline with yttrium and terbium ions. The present paper described how the complexes are prepared. Their IR absorption spectra, thermal analysis and fluorescence spectra are studied.

## 2 Experiments

### 2.1 Reagents and Instruments

The purity of rare earth oxides  $Tb_4O_7$  and  $Y_2O_3$  are all 99.99%; the purity of phenoxyacetic acid is 98.5%; 1,10-phenanthroline, anhydrous alcohol and other reagents are all analytical grade.

The determinations of C, H and N were performed by a Perkin-Elmer 2400 elemental analysis

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instrument and the contents of rare earth elements were obtained by EDTA titration. The IR spectra of the complexes were recorded on a Nicolet Nexus 670 FT-IR spectrophotometer in a form of KBr pellet. Molar conductivity measurements were made by using a  $10^{-3}$  mol · L<sup>-1</sup> solutions in DMF on a DDS-11A conductometer at 25 °C. The thermal analysis data were recorded on a Shimadzu DT-40 thermal analysis instrument with  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> as reference, the heating rate was 10 °C · min<sup>-1</sup>. The ultraviolet absorption spectra were taken with a TU-1901 spectrophotometer. The fluorescent excitation and emission spectra were obtained on a Shimadzu RF-5301PC fluorescence photometer, the exbandpass and embandpass were all 1.5 nm. Fluorescence lifetime measurements were carried out on an Edinburgh FLS920 phosphorimeter.

## 2.2 Synthesis of Complexes

### 2.2.1 Preparation of ethanol solutions of TbCl<sub>3</sub> and YCl<sub>3</sub>

The rare-earth oxide (Y<sub>2</sub>O<sub>3</sub>) was dissolved in diluted hydrochloric acid (HCl), and the surplus HCl was removed by evaporation. The residue was dissolved with anhydrous alcohol. Otherwise, H<sub>2</sub>O<sub>2</sub> was increased when prepared the anhydrous alcohol solution of TbCl<sub>3</sub> with Tb<sub>4</sub>O<sub>7</sub>.

### 2.2.2 Synthesis of rare earth complexes

A mixed solution of TbCl<sub>3</sub> and YCl<sub>3</sub> [ $n(\text{Tb}^{3+}) : n(\text{Y}^{3+}) = 0.10 : 0.90 ; 0.20 : 0.80 ; 0.30 : 0.70 ;$

$0.40 : 0.60 ; 0.60 : 0.40 ; 0.70 : 0.30 ; 0.80 : 0.20 ; 0.90 : 0.10$ ] was added in the beaker. For the synthesis of the Tb<sub>x</sub>Y<sub>1-x</sub>(POA)<sub>3</sub>phen complexes, phenoxycetic acid and 1, 10-phenanthroline was dissolved in anhydrous alcohol in 25 mL circularly flask and the anion was formed by addition of a solution of NaOH to pH ≈ 6 ~ 7. A mixed solution of TbCl<sub>3</sub> and YCl<sub>3</sub> was added dropwise to this solution and the reacting mixture solution was refluxed about 3 h.

The white precipitates Tb<sub>x</sub>Y<sub>1-x</sub>(POA)<sub>3</sub>phen · 1/2H<sub>2</sub>O were formed in the reaction mixture solutions, cooled the mixtures to room temperature and filtered them. The products were washed with anhydrous alcohol and dried to constant weight.

## 3 Results and Discussion

### 3.1 Composition Analysis and Molar Conductivities

The elemental analysis data of C, H, N and EDTA titration of the complexes show that the composition of the complexes conforms to chemical formula Tb<sub>x</sub>Y<sub>1-x</sub>(POA)<sub>3</sub>phen · 1/2H<sub>2</sub>O ( $x = 1.0, 0.9, 0.8, 0.7, 0.6, 0.5, 0.4, 0.3, 0.2, 0.1$ ). The molar conductivities range of all the complexes were in 10.0 ~ 13.0 S · cm<sup>2</sup> · mol<sup>-1</sup> in DMF reagent. The results show that complexes are non-electrolyte<sup>[13,14]</sup>. The theoretical and measured values of the complexes are shown in Table 1. All the complexes are white powders. They can be dissolved in DMF and DMSO.

**Table 1** Composition analysis (%) and molar conductivities (S · cm<sup>2</sup> · mol<sup>-1</sup>) of the complexes

Complexes	C	H	N	RE	$\lambda_m$
	Found (Calc)	Found (Calc)	Found (Calc)	Found (Calc)	Found
Tb <sub>0.1</sub> Y <sub>0.9</sub> L <sub>3</sub> L' · 1/2H <sub>2</sub> O	58.13(58.51)	3.85(4.06)	3.59(3.79)	12.54(12.99)	13.0
Tb <sub>0.2</sub> Y <sub>0.8</sub> L <sub>3</sub> L' · 1/2H <sub>2</sub> O	58.27(57.96)	4.41(4.02)	3.67(3.75)	13.52(13.81)	11.4
Tb <sub>0.3</sub> Y <sub>0.7</sub> L <sub>3</sub> L' · 1/2H <sub>2</sub> O	57.93(57.47)	3.85(3.98)	3.58(3.72)	14.34(14.61)	12.7
Tb <sub>0.4</sub> Y <sub>0.6</sub> L <sub>3</sub> L' · 1/2H <sub>2</sub> O	56.72(56.89)	3.97(3.95)	3.67(3.68)	15.76(15.58)	12.4
Tb <sub>0.5</sub> Y <sub>0.5</sub> L <sub>3</sub> L' · 1/2H <sub>2</sub> O	56.47(56.37)	3.80(3.91)	3.56(3.65)	16.45(16.18)	10.0
Tb <sub>0.6</sub> Y <sub>0.4</sub> L <sub>3</sub> L' · 1/2H <sub>2</sub> O	56.01(55.86)	3.93(3.87)	3.53(3.61)	16.63(16.93)	12.2
Tb <sub>0.7</sub> Y <sub>0.3</sub> L <sub>3</sub> L' · 1/2H <sub>2</sub> O	55.18(55.36)	3.94(3.85)	3.37(3.58)	17.50 (17.67)	12.0
Tb <sub>0.8</sub> Y <sub>0.2</sub> L <sub>3</sub> L' · 1/2H <sub>2</sub> O	55.10(54.87)	3.96(3.81)	3.56 (3.55)	18.60 (18.40)	11.7
Tb <sub>0.9</sub> Y <sub>0.1</sub> L <sub>3</sub> L' · 1/2H <sub>2</sub> O	54.62(54.39)	4.00(3.77)	3.53(3.52)	18.88 (19.12)	12.5
TbL <sub>3</sub> L' · 1/2H <sub>2</sub> O	53.69(53.91)	3.72 (3.74)	3.22(3.49)	20.17(19.83)	11.0

(L = C<sub>6</sub>H<sub>5</sub>OCH<sub>2</sub>COO<sup>-</sup>, L' = phen)

### 3.2 TG-DTA Curves

The determination range of temperature is 30 ~ 700 °C. The results show that TG-DTA curves of the complexes are all similar. For the complex TbL<sub>3</sub>L' · 1/2H<sub>2</sub>O, which present an endothermic peak at 274.7 °C and the mass loss of 1.20%, which corresponds very well to release half water content (1.14%). The relatively high temperature of water loss shows that they are coordinated water. The result is the same as those of elemental analysis and IR absorption spectra. One exothermic peak is observed, it appears at 576.2 °C, indicating that the complexes are stable below 576 °C, when the temperature rise to over 576 °C, the decomposition and oxidization occurs.

### 3.3 IR Absorption Spectra

The IR spectra data for HPOA, Phen and the complexes are shown in Table 2. From Table 2, the

**Table 2 Major IR spectra data of ligands and the complexes with different Tb<sup>3+</sup>:Y<sup>3+</sup> proportion** cm<sup>-1</sup>

Ligands and complexes	$\nu_{\text{O-H}}$	$\nu_{\text{C=O}}(\text{COOH})$	$\nu_{\text{as}}(\text{COO}^-)$	$\nu_{\text{s}}(\text{COO}^-)$	$\nu_{\text{C-O}}$	$\nu_{\text{C=N}}$	$\delta_{\text{O-H}}(\text{COOH})$	$\delta_{\text{C-H}}$
Phen	—	—	—	—	—	1 586	—	853/739
HPOA	3 033/2 575	1 735	1 592	1 436	1 233	—	1 093	—
Tb <sub>0.1</sub> Y <sub>0.9</sub>	3 426	—	1 636	1 429	1 221	1 516	—	845/727
Tb <sub>0.2</sub> Y <sub>0.8</sub>	3 425	—	1 636	1 429	1 221	1 517	—	845/727
Tb <sub>0.3</sub> Y <sub>0.7</sub>	3 427	—	1 636	1 429	1 221	1 517	—	845/727
Tb <sub>0.4</sub> Y <sub>0.6</sub>	3 424	—	1 635	1 429	1 221	1 517	—	845/727
Tb <sub>0.6</sub> Y <sub>0.4</sub>	3 424	—	1 634	1 429	1 221	1 516	—	845/727
Tb <sub>0.7</sub> Y <sub>0.3</sub>	3 426	—	1 634	1 429	1 221	1 516	—	845/727
Tb <sub>0.8</sub> Y <sub>0.2</sub>	3 425	—	1 634	1 429	1 221	1 517	—	845/727
Tb <sub>0.9</sub> Y <sub>0.1</sub>	3 425	—	1 633	1 428	1 221	1 516	—	845/727

C=N and C—H stretching vibration peaks appear at 1561, 739.2 and 853.1 cm<sup>-1</sup> in 1,10-phenanthroline, and they shift to about 1 536, 729 and 847 cm<sup>-1</sup> in the complexes. It indicates that the chemical bonds are formed between rare earth ions and nitrogen atoms of 1,10-phenanthroline<sup>[15]</sup>.

In addition, in the ternary complexes, there are absorption bands of  $\nu_{\text{O-H}}$  from 3 100 to 3 500 cm<sup>-1</sup>, it is considered as  $\nu_{\text{O-H}}$  of water. It shows that the ternary complexes contain water molecules.

### 3.4 UV Absorption Spectra

The UV absorption spectra of the ligands and

complexes are obviously different in comparison with two ligands HPOA and phen. It shows that rare earth ions are bonded with the ligands. The IR absorption spectra of the complexes are similar indicating their structures are similar.

After the complexes are formed, the absorption peak of  $\nu_{\text{COOH}}$  disappears. In the complexes the asymmetric stretching vibrations of carboxylate group appears in the region of 1 635 cm<sup>-1</sup> and the symmetric stretching vibrations appears in the region 1 429 cm<sup>-1</sup>. The stretching vibration of  $\nu_{\text{C-O}}$  appear in the region of 1 221 cm<sup>-1</sup>. The stretching vibration peak of the carboxyl group is located at 1 735 cm<sup>-1</sup> and the  $\delta_{\text{OH-}}$  with  $\nu$  coincidence vibration gives absorption at 1 093 cm<sup>-1</sup> for phenoxyacetic acid. These facts show that the coordinate bonds are formed between oxygen atoms in the phenoxyacetate with the rare earth ions<sup>[15]</sup>.

complexes are measured in DMF within 200 ~ 400 nm region. Fig. 1 shows the UV absorption spectra

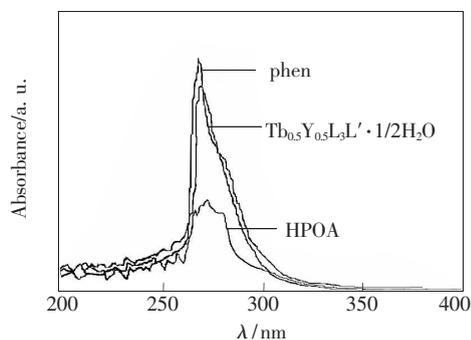


Fig. 1 UV absorption spectra of Phen, HPOA and Tb<sub>0.5</sub>Y<sub>0.5</sub>-L<sub>3</sub>L' · 1/2H<sub>2</sub>O.

of HPOA, Phen and  $\text{Tb}_{0.5}\text{Y}_{0.5}(\text{POA})_3\text{phen} \cdot 1/2\text{H}_2\text{O}$ .

From the ultraviolet spectra, we can see that the dissociative ligands and the complexes have absorption in ultraviolet radiation. UV absorption of HPOA attach to the major  $\pi\text{-}\pi^*$  electronic transitions in 270.0 nm and those of Phen attach to the major  $\pi\text{-}\pi^*$  electronic transitions in 264 nm. The UV absorption spectra for all complexes are similar. Comparing the absorption spectra of HPOA, Phen and complexes, a blue-shift from 270 to 266 nm and a red-shift from 264 to 266 nm of the major  $\pi\text{-}\pi^*$  electronic transitions are observed. These facts show that the coordinated bonds are formed between oxygen atoms of HPOA and nitrogen atoms of 1, 10-phenanthroline with the rare earth ions in the

complexes

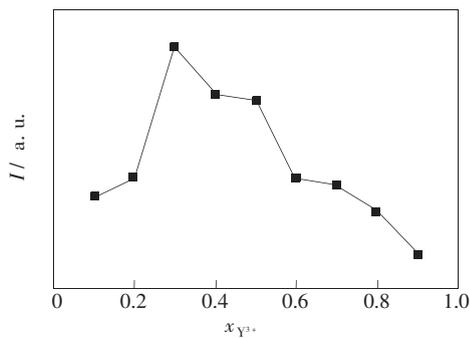
### 3.5 Fluorescence Spectra

At the room temperature, under the condition of the entrance and the exit slits are both 1.5 nm wide, by taking the fluorescence emission wavelength of terbium, *i. e.*, 545 nm, as the monitoring wavelength, the excitation spectra are wide bond peaks and the spectra are all similar, of which the best excited wavelength is 331 nm. It shows that the lowest excitation state energy level of  $\text{Tb}^{3+}$  ion with the triplet state energy of the lignd phenoxyacetic acid and 1, 10-phenanthroline matches well each other, the absorbing energy of the ligands may effectively transfers to  $\text{Tb}^{3+}$  ion. The emission spectra intensities of the complexes excited by 331 nm are given in Table 3.

**Table 3** Fluorescent spectra data of the complexes with different ( $\text{Tb}^{3+}:\text{Y}^{3+}$ ) proportion

Complexes	$\lambda_{\text{em}}/\text{nm}$	$I/\text{a. u}$						
$\text{Tb}_{0.1}\text{Y}_{0.9}$	489.6	62.01	545.4	114.80	584.6	10.18	621.2	7.68
$\text{Tb}_{0.2}\text{Y}_{0.8}$	489.6	89.03	545.4	165.10	584.6	14.55	621.0	11.76
$\text{Tb}_{0.3}\text{Y}_{0.7}$	489.6	103.37	545.4	196.95	584.8	16.76	621.4	7.42
$\text{Tb}_{0.4}\text{Y}_{0.6}$	489.6	107.83	545.4	203.05	584.6	17.26	621.2	7.10
$\text{Tb}_{0.5}\text{Y}_{0.5}$	489.6	149.92	545.4	294.73	585.0	25.25	621.2	13.58
$\text{Tb}_{0.6}\text{Y}_{0.4}$	489.6	159.93	545.4	302.64	585.0	25.87	621.4	11.40
$\text{Tb}_{0.7}\text{Y}_{0.3}$	489.6	183.78	545.4	357.56	584.8	30.34	621.2	14.68
$\text{Tb}_{0.8}\text{Y}_{0.2}$	489.6	109.20	545.4	206.28	584.6	18.64	621.0	18.50
$\text{Tb}_{0.9}\text{Y}_{0.1}$	489.6	96.92	545.2	182.91	584.8	16.01	621.2	12.27

As seen from Table 3, there are four emission spectra peaks about 489.6, 545.4, 584.6, 621.2 nm which contribute to the transition of  ${}^5\text{D}_4 \rightarrow {}^7\text{F}_6$ ,  ${}^5\text{D}_4 \rightarrow {}^7\text{F}_5$ ,  ${}^5\text{D}_4 \rightarrow {}^7\text{F}_4$ ,  ${}^5\text{D}_4 \rightarrow {}^7\text{F}_3$  of  $\text{Tb}^{3+}$ , and among them the  ${}^5\text{D}_4 \rightarrow {}^7\text{F}_5$  transition is the strongest. The fluorescence peak positions for the complexes do not change obviously with doping quantity of  $\text{Y}^{3+}$ , but the fluorescence emission intensities change obviously. The data of the fluorescence spectra of the complexes are enhanced when  $\text{Y}^{3+} = 0.5, 0.4, 0.3$  and the fluorescence intensity of  $\text{Tb}_{0.7}\text{Y}_{0.3}(\text{POA})_3\text{phen} \cdot 1/2\text{H}_2\text{O}$  has the strongest fluorescence intensity among the complexes. So, we can make such a conclusion; when inert rare earth ion  $\text{Y}^{3+}$  is added to the pure complex at an appropriate ratio, the intrinsic emission peaks of  $\text{Tb}^{3+}$  are much enhanced. The mechanism of this co-luminescence enhancing effect



**Fig. 2** Emission intensity ( ${}^5\text{D}_4 \rightarrow {}^7\text{F}_5$ ) of the complexes versus content of  $\text{Y}^{3+}$  ions

is as follows: POA- and 1, 10-phenanthroline can absorb radiant energy and then transfer it to  $\text{Tb}^{3+}$ , the intramolecular energy transfer occurs and the transfer process from the energy donor to  $\text{Tb}^{3+}$  is enhanced, which agrees with the similar phenomenon in other systems<sup>[16]</sup>.

### 3.6 Fluorescent Lifetime

The decay curve of  $TbL_3L' \cdot 1/2H_2O$  and  $Tb_{0.5}Y_{0.5}L_3L' \cdot 1/2H_2O$  are measured with the excitation wavelength at 340 nm and the emission wavelength at 545 nm at room temperature. The  $^5D_4$  excited level lifetime of  $Tb^{3+}$  in the both complexes is 631.8  $\mu s$  ( $x^2 = 1.020$ ) and 696.2  $\mu s$  ( $x^2 = 1.197$ ), respectively. The lifetime of  $Tb_{0.5}Y_{0.5}L_3L' \cdot 1/2H_2O$  is longer than that of  $TbL_3L' \cdot 1/2H_2O$ . It is clear that the fluorescence of  $Tb^{3+}$  in the both complexes shows simple decay behavior. The decay curve fitted well into a singly exponential function as  $I = I_0 \exp(-t/\tau)$  ( $\tau$  is  $1/e$  lifetime of the rare earth ion), indicating that all the  $Tb^{3+}$  ions occupy the same average coordination environment. The fluorescence lifetime of the both complexes exhibits the similar feature.

## 4 Conclusion

A series of doped complexes with different molar ratio of  $Tb^{3+}$  to  $Y^{3+}$  with phenoxyacetic acid and 1, 10-phenanthroline were synthesized. Elemental analysis and rare earth complexometric titration showed that the composition of the complexes are  $Tb_xY_{1-x}(POA)_3 \text{ phen} \cdot 1/2H_2O$ . The fluorescence emission intensities change obviously with doping quantity of  $Y^{3+}$ . The data of the fluorescence spectra of complexes enhance as  $Y^{3+} = 0.5, 0.4, 0.3$ , and the fluorescence intensity of  $Tb_{0.7}Y_{0.3}(POA)_3 \text{ phen} \cdot 1/2H_2O$  is the strongest among the complexes. It shows that the intramolecular energy transfer occurs and the transfer process from the energy donor to  $Tb^{3+}$  is efficient.

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## 苯氧乙酸、邻菲罗啉铽-钇配合物的合成、表征及荧光性能

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**摘要:** 以苯氧乙酸和邻菲罗啉为配体, 不同比例铽钇为中心, 在无水乙醇中合成了一系列铽掺钇配合物。元素分析和稀土络合滴定推测配合物的组成为  $\text{Tb}_x\text{Y}_{1-x}(\text{POA})_3 \text{phen} \cdot 1/2\text{H}_2\text{O}$  ( $\text{POA}^- = \text{C}_6\text{H}_5\text{OCH}_2\text{COO}^-$ ,  $x = 1.0, 0.9, 0.8, 0.7, 0.6, 0.5, 0.4, 0.3, 0.2, 0.1$ )。红外光谱测试表明, 苯氧乙酸的羧基氧及苯氧基氧与稀土离子配位。邻菲罗啉的两个氮原子也与稀土离子配位; 热分析表明: 该系列配合物在 274 °C 附近失去配位水, 温度高于 576 °C 发生氧化分解。荧光光谱测试结果表明: 该系列配合物都可发出较强的特征荧光, 在一定比例范围内, 钇可以增强铽的发光, 组成为配合物  $\text{Tb}_{0.7}\text{Y}_{0.3}(\text{POA})_3 \text{phen} \cdot 1/2\text{H}_2\text{O}$  荧光最强; 荧光寿命与荧光强度变化一致。

**关键词:** 铽掺钇配合物; 苯氧乙酸; 邻菲罗啉; 荧光性质

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