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# Crystal structure and properties of complex [Eu(m-BrBA)<sub>3</sub>(H<sub>2</sub>O)(phen)]<sub>2</sub>

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#### Abstract

A binuclear europium complex  $[Eu(m-BrBA)_3(H_2O)(phen)]_2$  (1) has been obtained in the ethanol solution and characterized structurally by X-ray diffraction, where m-BrBA = m-bromobenzoate and phen = 1,10-phenanthroline. Each Eu<sup>3+</sup> ion is eight-coordinated to one 1,10-phenanthroline molecule, one monodentate carboxylate group, one water molecule and four bridging carboxylate groups in which the carboxylate groups are bonded to the europium ion in two modes: the monodentate and the bridging bidentate. Excitation and luminescence data observed at room temperature show that the title complex emits red fluorescence under ultraviolet light. The results of thermal analysis indicate that the complex  $[Eu(m-BrBA)_3(H_2O)(phen)]_2$  is quite stable to heat.

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Keywords: Europium complex; m-Bromobenzoic acid; 1,10-Phenanthroline; Crystal structure; Luminescence

## 1. Introduction

The rare-earth carboxylic acid complexes have many special structures and interesting spectroscopic properties, which are of great interest in extraction, separation, germicide preparation, catalysis, luminescence and functional material preparation. Therefore the study of them has been a rich topic for chemical workers all through [1]. Structural studies of ternary europium complexes with aromatic acids and nitrogen-containing ligands have shown that the rare earth atoms have several types of coordination, usually with coordination number of eight or nine, of which there is no coordinated water molecules. These complexes result in strong emission of a red colour and are stable to light and heat [2-10]. As part of our continuing study of europium carboxylate complexes, the present paper reports the crystal structure, luminescence spectra and thermal analysis of a special complex  $[Eu(m-BrBA)_3(H_2O)(phen)]_2$ , in which there are two coordinated water molecules.

## 2. Experimental

## 2.1. General

All the starting materials were of analytical grade. Excitation and luminescence spectra were

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performed at room temperature using a MPF-4 fluorescence spectrophotometer. The TG and DTG experiment for the title complex were performed using a Perkin–Elmer TGA7 thermogravimetric analyzer. The heating rate was 10 °C min<sup>-1</sup> from ambient to 918 °C under an air atmosphere. The sample weight was 2.562 mg.

#### 2.2. Preparation of $[Eu(m-BrBA)_3(H_2O)(phen)]_2(1)$

A stoichiometric amount of EuCl<sub>3</sub>·6H<sub>2</sub>O, mbromobenzoic acid and 1,10-phenanthroline were dissolved in 95% C<sub>2</sub>H<sub>5</sub>OH, respectively. The pH of the m-bromobenzoic acid was adjusted to the range of 6-7 with 1 M NaOH solution. The C<sub>2</sub>H<sub>5</sub>OH solutions of two ligands were mixed and then the mixture was added dropwise to the ethanolic EuCl<sub>3</sub> solution, while a white precipitate formed. Crystals suitable for X-ray investigation were obtained from the mother liqour

Table 1

Data collection and processing parameters for complex (1)

after a month. Anal. Calcd. (%) for  $C_{66}H_{44}N_4O_{14}Br_{6-}$ Eu<sub>2</sub>: C 41.38(41.71), H 2.27(2.33), N 3.06(2.95).

#### 3. Structure determination

Determination of the unit cell and data collection was performed on a CCD area detector using graphite monochromated Mo K $\alpha$  radiation ( $\lambda = 0.71073$  Å) at 293(2) K. A total of 8009 [ $R_{int} = 0.1193$ ] independent reflections were collected by  $\omega$ -2 $\theta$  scan technique in the range 2.01°  $\leq \theta \leq 25.00^{\circ}$  from which 5580 [ $I > 2\sigma(I)$ ] reflections were corrected. The structure was solved using direct methods in SHELXS-97 [11] and refined using a full-matrix leastsquare procedure on  $F^2$  in SHELXL-97 [12]. All nonhydrogen atoms were refined with anisotropic thermal parameters, hydrogen atoms were added theoretically and refined with riding model position parameters and fixed isotropic thermal parameters.

Empirical formula	$C_{66}H_{44}N_4O_{14}Br_6Eu_2$	
Formula weight	1900.43	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group $P\bar{1}$		
Unit cell dimensions	a = 8.1061(10)  Å	
	$\alpha = 94.200(2)^{\circ}$	
	b = 12.9770(15) Å	
	$\beta = 94.780(2)^{\circ}$	
	c = 15.5431(18)Å	
	$\gamma = 79.903(2)^{\circ}$	
Volume	1601.2(3)Å <sup>3</sup>	
Z, Calculated density	$1, 1.971 \text{ Mg/m}^3$	
Absorption coefficient 5.753 mm <sup>-1</sup>		
F(00) 916		
$\theta$ range for data collection	$2.01-25.00^{\circ}$	
Limiting indices	$-9 \le h \le 8, -14 \le k \le 15, -18 \le l \le 17$	
Reflections collected/unique	8009/5580 [R(int) = 0.1193]	
Completeness to theta $= 25.00$	98.8 %	
Absorption correction	Sadabs	
Max and min transmission	1.00000 and 0.50964	
Refinement method	Full-matrix least-squares on $F^2$	
Data/restraints/parameters	5580/0/415	
Goodness-of-fit on $F^2$	1.068	
Final <i>R</i> indices $[I > 2\sigma(I)]$ $R1 = 0.0747, wR2 = 0.1920$		
<i>R</i> indices (all data) $R1 = 0.0894, wR2 = 0.2015$		
Largest diff. Peak and hole	2.737 and $-2.843 \text{ e}\text{\AA}^{-3}$	

	X	Y	Ζ	U(eq)
Eu	7994(1)	1316(1)	5187(1)	22(1)
Br(1)	8088(3)	4668(2)	9143(1)	82(1)
Br(2)	11048(4)	-3506(2)	8585(2)	129(1)
Br(3)	6469(3)	1171(2)	319(1)	79(1)
O(1)	6730(10)	2083(6)	6442(5)	39(2)
O(2)	4180(12)	1668(8)	6570(6)	54(3)
O(3)	7576(10)	-267(7)	5749(5)	39(2)
O(4)	9800(10)	-1529(7)	5660(5)	40(2)
O(5)	7449(10)	550(6)	3769(5)	38(2)
O(6)	9611(10)	-713(6)	3952(5)	35(2)
O(7)	5001(10)	1310(6)	4958(5)	39(2)
N(1)	6775(12)	2926(7)	4323(7)	35(2)
N(2)	9025(13)	3079(7)	5712(7)	39(2)
C(1)	5790(18)	2844(11)	3595(9)	50(3)
C(2)	5060(20)	3714(14)	3121(11)	68(5)
C(3)	5380(20)	4662(15)	3407(12)	73(5)
C(4)	6374(18)	4808(11)	4187(10)	54(4)
C(5)	6730(19)	5847(10)	4576(14)	68(5)
C(6)	7680(20)	5913(11)	5299(14)	68(5)
C(7)	8519(17)	4953(10)	5720(11)	51(4)
C(8)	9588(18)	5002(12)	6465(12)	61(4)
C(9)	10380(20)	4092(13)	6792(11)	63(4)
C(10)	10105(17)	3161(10)	6406(8)	43(3)
C(11)	8221(15)	3966(9)	5352(9)	39(3)
C(12)	7101(15)	3908(10)	4616(10)	45(3)
C(13)	5423(15)	2071(9)	6852(8)	36(3)
C(14)	5397(15)	2606(9)	7751(8)	35(3)
C(15)	6575(16)	3240(10)	8004(8)	41(3)
C(16)	6513(18)	3767(10)	8790(8)	43(3)
C(17)	5260(20)	3673(12)	9347(9)	59(4)
C(18)	4110(20)	3079(12) 3079(14)	9078(10)	68(5)
C(19)	4167(19)	2522(12)	8282(9)	52(3)
C(20)	8610(14)	-1028(8)	60202(7)	30(2)
C(21)	8380(16)	-1380(11)	6024(8)	$\frac{30(2)}{41(3)}$
C(21)	9564(19)	-2172(11)	7259(8)	48(3)
C(22)	9304(19)	-2461(14)	8077(10)	+0(3) 67(5)
C(23)	8180(40)	-1960(20)	8584(12)	103(0)
C(24)	7070(40)	-1200(20)	8230(12)	113(10)
C(25)	7070(40)	-868(14)	7385(10)	65(A)
C(20)	7070(20) 8520(15)	-308(14) -150(0)	2476(8)	$\frac{03(4)}{27(2)}$
C(27)	8530(13)	-324(10)	3470(8)	37(3)
C(28)	8040(14) 7622(16)	= 324(10)	2307(7)	32(3)
C(29)	7055(10)	220(10)	1950(8)	39(3)
C(30)	7750(18)	220(10)	1009(8)	40(3)
C(31)	0020(20)	-023(12) -1251(12)	147(9)	55(4) 60(4)
C(32)	9930(20)	-1231(12)	12/0(10)	51(2)
U(33)	9847(17) 5577	-1104(11)	2157(10)	51(5)
П(1)	3377	2184	3394	00
H(2)	43/3	5032	2621	81
H(5)	4944	5238	3087	8/
H(5)	6268	6455	4302	81
H(6)	/836	6568	5546	82

Table 2 (continued)	

	(commueu)			
	X	Ŷ	Z	U(eq)
H(8)	9752	5645	6732	74
H(9)	11123	4106	7282	75
H(10)	10692	2548	6633	51
H(15)	7400	3304	7640	49
H(17)	5232	4021	9892	71
H(18)	3259	3036	9432	82
H(19)	3376	2095	8111	62
H(22)	10421	-2511	6925	57
H(24)	8094	-2161	9140	123
H(25)	6221	-865	8578	136
H(26)	6258	- 343	7159	78
H(29)	6876	916	2184	47
H(31)	9033	-738	154	66
H(32)	10696	-1803	1049	72
H(33)	10600	- 1528	2521	61

Further details of the structure analysis are given in Table 1. Positional parameters and selected bond lengths and angles are presented in Tables 2 and 3, respectively.

#### 4. Results and discussion

#### 4.1. Crystal structure of $[Eu(m-BrBA)_3(H_2O)(phen)]_2$

The molecular structure and atomic numbering of the complex are shown in Fig. 1. The two  $Eu^{3+}$  ions are linked by four carboxylate groups through their bidentate bridging modes, form a dimeric unit with crystallographic inversion center. Each Europium ion is coordinated to eight atoms, of which four oxygen atom are from the bridging carboxylates, one oxygen atom from the monodentate carboxylate group, one oxygen atom from the water molecular and two nitrogen atoms from a 1,10-phenanthroline molecule.

In the binuclear molecule, the Eu–O distances are in a range of 2.315(8)-2.424(8) Å, in which the Eu–O bonds formed by water molecules are the longest. The Eu–O bond distances formed by the bridging carboxylates are slightly longer than that formed by the monodentate carboxylate groups except for Eu–O(6)#1[Eu#1–O(6)]. The average bond distances of Eu–O and Eu–N are 2.372 and 2.598 Å, respectively, which slightly shorter than Table 3

Selected bond lengths (Å) and angles (°) for complex (1) (Symmetry transformations used to generate equivalent atoms: #1 -x + 2, -y, -z + 1)

Eu-O(6)#1 2.315(8) Eu-O(4)#1 2.372(8)	Eu-O(1)	2,352(8)
Eu-O(4)#1 2.372(8)		2.002(0)
	Eu-O(3)	2.378(8)
Eu–O(5) 2.391(8)	Eu-O(7)	2.424(8)
Eu–N(1) 2.572(9)	Eu-N(2)	2.623(9)
Eu-Eu#1 4.3261(11)	Br(1)–C(16)	1.900(14)
Br(2)-C(23) 1.858(19)	Br(3)-C(30)	1.875(14)
O(1)-C(13) 1.284(14)	O(2)-C(13)	1.248(15)
O(3)-C(20) 1.253(14)	O(4)-C(20)	1.220(13)
O(4)-Eu#1 2.372(8)	O(5)-C(27)	1.242(14)
O(6)-C(27) 1.258(15)	O(6)-Eu#1	2.315(8)
N(1)-C(1) 1.337(17)	N(1)-C(12)	1.384(16)
N(2)-C(10) 1.341(17)	N(2)- C(11)	1.353(16)
O(6)#1-Eu-O(1) 88.6(3)	O(6)#1-Eu-O(4)#1	76.8(3)
O(1)-Eu-O(4)#1 139.0(3)	O(6)#1-Eu-O(3)	72.9(3)
O(1)-Eu-O(3) 83.1(3)	O(4)#1-Eu-O(3)	126.6(3)
O(6)#1-Eu-O(5) 122.0(3)	O(1)–Eu–O(5)	144.0(3)
O(4)#1-Eu-O(5) 72.2(3)	O(3)–Eu–O(5)	88.1(3)
O(6)#1-Eu-O(7) 144.0(3)	O(1)–Eu–O(7)	72.1(3)
O(4)#1-Eu-O(7) 136.9(3)	O(3)-Eu-O(7)	74.7(3)
O(5)-Eu-O(7) 71.9(3)	O(6)#1-Eu-N(1)	138.5(3)
O(1)- Eu-N(1) 92.2(3)	O(4)#1-Eu-N(1)	75.7(3)
O(3)- Eu-N(1) 148.2(3)	O(5)-Eu-N(1)	77.4(3)
O(7)–Eu–N(1) 73.9(3)	O(6)#1-Eu-N(2)	78.8(3)
O(1)-Eu-N(2) 68.2(3)	O(4)#1-Eu-N(2)	71.4(3)
O(3)-Eu-N(2) 139.9(3)	O(5)-Eu-N(2)	131.5(3)
O(7)-Eu-N(2) 118.8(3)	N(1)-Eu-N(2)	63.3(3)
O(6)#1-Eu-C(27) 101.9(3)	O(1)-Eu-C(27)	158.4(3)
O(4)#1-Eu-C(27) 62.4(3)	O(3)-Eu-C(27)	82.1(3)
O(5)-Eu-C(27) 20.1(3)	O(7)-Eu-C(27)	88.9(3)
N(1)-Eu-C(27) 92.2(3)	N(2)-Eu-C(27)	132.0(3)
O(6)#1-Eu-Eu#1 48.5(2)	O(1)-Eu-Eu#1	131.4(2)
O(4)#1-Eu-Eu#1 61.8(2)	O(3)-Eu-Eu#1	65.2(2)
O(5)-Eu-Eu#1 73.57(19)	O(7)-Eu-Eu#1	127.0(2)
N(1)-Eu-Eu#1 133.9(2)	N(2)-Eu-Eu#1	114.2(2)
C(27)-Eu-Eu#1 53.6(2)	C(13)-O(1)-Eu	140.0(7)
C(20)– O(3)– Eu 130.9(7)	C(20)-O(4)-Eu#1	141.6(8)
C(27)–O(5)–Eu 118.4(8)	C(27)-O(6)-Eu#1	163.1(8)
C(1)–N(1)–Eu 121.4(8)	C(12)-N(1)-Eu	120.1(8)
C(10)–N(2)–Eu 122.7(8)	C(11)-N(2)-Eu	117.7(8)
N(2)-C(11)-C(12) 120.3(10)	N(1)-C(12)-C(11)	117.4(11)
O(2)-C(13)-O(1) 125.7(11)	O(2) - C(13) - C(14)	117.8(10)
O(1)-C(13)-C(14) 116.5(10)	O(4)-C(20)-O(3)	127.5(11)
O(4)-C(20)-C(21) 115.3(11)	O(3) - C(20) - C(21)	117.2(11)
O(5)-C(27)-O(6) 122.8(11)	O(5)-C(27)-C(28)	119.3(11)
O(6)-C(27)-C(28) 117.8(11)	O(5)-C(27)-Eu	41.5(6)
O(6)-C(27)-Eu 83.8(7)	C(28)– C(27)– Eu	151.7(8)

corresponding average distances 2.389 and 2.599 Å found in the complex  $[Eu(m-MBA)_3phen]_2 \cdot (H_2O)_2$ [2]. The fact indicates that the interaction of europium ion with oxygen atom or nitrogen atom in

 $[Eu(m-MBA)_3phen]_2 \cdot (H_2O)_2$  is weaker than that in the complex  $[Eu(m-BrBA)_3(H_2O)(phen)]_2$ . Compared with the analogue complexes with aromatic acids and 1,10-phenanthroline ligands [2-6], only the crystal



Fig. 1. The molecular structure of complex (1).

for the title complex and  $[Eu(m-MBA)_3phen]_2 \cdot 2H_2O$ contain water molecules, and water molecules are not coordinated in the complex  $[Eu(m-MBA)_3phen]_2$ .  $2H_2O$ . Because of the coordination effect of water molecules, the unbridging bidentate carboxylate groups become monodentate in the title complex. These are possibly caused by the electronic effect of different substituent on the benzene ring and the steric effect.

In the carboxylate groups, C–O distances differ significantly; coordination causes lengthening of C–O[1.284(14) Å] for the monodentate carboxylate groups in comparison to the bidentate-bridging carboxylate groups[1.220(13)–1.258(15) Å]. The O(1)–C(13)–O(2) angle in the monodentate

carboxylate group is  $125.7(11)^\circ$ . The O(3)–C(20)–O(4) [O(5)–C(27)–O(6)] angle in the bidentate bridging carboxylate group is  $127.5(11)^\circ$  [122.8(11)°]. The five atoms of the chelated ring containing two chelated nitrogen atoms and europium ion are coplanar in the dimer since the molecule of 1,10-phenanthroline is rigid.

#### 4.2. Thermal decomposition

The TG and DTG curves of the  $[Eu(m-BrBA)_3(-H_2O)(phen)]_2$  are shown in Fig. 2. The thermoanalytical data for the complex are given in Table 4. The Eu(III) complex decomposed via intermediates to give the europium oxide as an end product. The percentage



Fig. 2. Thermogravimetric curves (DTG and TG) for complex (1).

of mass loss and probable composition of the expelled groups are also indicated in Table 4.

The results of thermal analysis indicate that title complex begins to decompose at the temperature of 107 °C and its decomposition ends at 918 °C. The TG degradation of [Eu(m-BrBA)<sub>3</sub>(H<sub>2</sub>O)(phen)]<sub>2</sub> reveals four decomposition stages, as predicted by the DTG curve(Fig. 2), and there are weight losses with maximum rate at 139, 381, 564 and 699 °C in

the DTG curve. The first stage start from 107 to 156 °C with a mass loss 2.15 wt% which corresponds to the loss of 2 mol H<sub>2</sub>O (theoretical loss is 1.90 wt%). The second stage degradation temperature is in the range of 264-464 °C with the mass loss of 23.28 wt%, in which 2 mol C<sub>12</sub>H<sub>8</sub>N<sub>2</sub> and 1 mol Br are removed with theoretical loss of 23.17 wt%. This degradation can be explained based on the bond distances of the structure (Table 3): Eu–N distance is longer than any

Table 4

Thermal decomposition data for comple	ex (1) in air atmosphere	from TG and DTG analysis
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Stage	Temperature range (°C)	DTG peak temperature (°C)	Loss of mass (wt%)		Probable composition	Intermediate
			TG	Theory	of expelled groups	
I	107-156	139	2.15	1.90	$2H_2O$	[Eu(m-BrBA) <sub>3</sub> (phen)] <sub>2</sub>
II	264-464	381	23.28	23.17	$2C_{12}H_8N_2 + Br$	$Eu_2(m-BrBA)_5(-C_6H_4COO)$
III	464-607	564	25.19	25.40	$5Br + 0.8(-C_6H_4CO)$	$Eu_2 (-C_6H_4COO)_{5,2}O_{0,8}$
IV	607-918	699	32.18 82.80 <sup>a</sup>	31.01 81.48	$5.2 (-C_6H_4COO) - 2.2O$	Eu <sub>2</sub> O <sub>3</sub>

<sup>a</sup> Tatal loss of mass.



Fig. 3. Luminescence spectrum of complex (1) (excited by 313 nm).

other bond distance, so the bond is less stable and easy to be broken down theoretically. The third stage start from 464 to 607 °C with a mass loss of 25.19 wt% which corresponds to the loss of 5 mol Br and  $0.8 \text{ mol}(-C_6H_4CO)$  (theoretical loss is 25.40 wt%). The degradation can also be demonstrated by the bond distances of the structure (Table 3): C(13)-O(1)distance is longer than any other C–O bond distance, so this bond is less stable and easy to be broken down. The fourthly stage degradation temperature is in the range of 607-918 °C with the mass loss of 32.18 wt%, in which  $[5.2 \text{ mol}(-C_6H_4COO)-2.2 \text{ mol } O]$  are removed with theoretical loss of 31.01 wt%. Up to now, water-1,10-phenanthroline-tris(m-bromobenzoate) europium(III) was completely degraded into  $Eu_2O_3$  with a total loss of 82.80 wt% (theoretical loss is 81.48 wt%). The thermal decomposition of [Eu(m- $BrBA_{3}(H_{2}O)(phen)]_{2}$  can be described as follow:

$$\begin{split} & [\text{Eu}(\text{m-BrBA})_3(\text{H}_2\text{O})(\text{phen})]_2 \rightarrow [\text{Eu}(\text{m-BrBA})_3 \\ & (\text{phen})]_2 \rightarrow \text{Eu}_2(\text{m-BrBA})_5(-\text{C}_6\text{H}_4\text{COO}) \rightarrow \text{Eu}_2 \\ & (-\text{C}_6\text{H}_4\text{COO})_{5,2}\text{O}_{0,8} \rightarrow \text{Eu}_2\text{O}_3 \end{split}$$

#### 4.3. Luminescence properties

The excitation and luminescence spectra of the title complex were recorded at room temperature.

The luminescence spectrum is shown in Fig. 3. The excitation of complex [Eu(m-BrBA)<sub>3</sub>(H<sub>2</sub>O) (phen)]<sub>2</sub> was effected in a range of 240–400 nm. The fluorescence was observed in a range of 550–650 nm by selective excitation at 313 nm. The emission spectrum composed of the characteristic emission peaks arising from the transition of Eu<sup>3+</sup>:  ${}^{5}D_{0} \rightarrow {}^{7}F_{0}(579 \text{ nm}), {}^{5}D_{0} \rightarrow {}^{7}F_{1}(589, 592, 595 \text{ nm}), {}^{5}D_{0} \rightarrow {}^{7}F_{2}(612, 618, 619, 620 \text{ nm}).$  The intensity of the luminescence band arising from the  ${}^{5}D_{0} \rightarrow {}^{7}F_{2}$  transition is most intense.

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