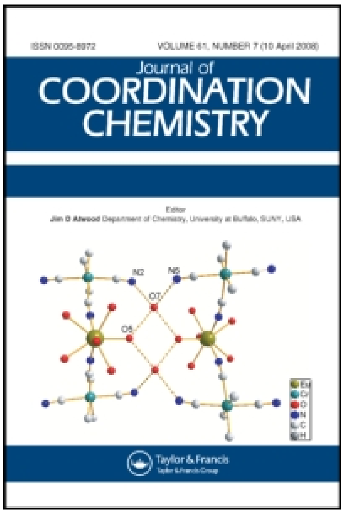

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Crystal Structure and Fluorescence Spectra of A Complex of Europium Picrate with *N,N,N′,N′*-tetraphenyl-3,6,9-trioxaundecanediamide

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CRYSTAL STRUCTURE AND FLUORESCENCE SPECTRA OF A COMPLEX OF EUROPIUM PICRATE WITH *N,N,N′,N′*-TETRAPHENYL-3,6,9-TRIOXAUNDECANEDIAMIDE

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The title complex Eu(Pic)$_3$(TTD) crystallizes in space group *Pcab* with $a = 23.714(4)$, $b = 29.245(6)$, $c = 32.035(8)\,\text{Å}$, $V = 22216(7)\,\text{Å}^3$, $Z = 16$, $D_c = 1.63\,\text{g cm}^{-1}$, $\mu = 12.5\,\text{cm}^{-1}$, $F(000) = 10976$ and its structure was solved and refined to $R = 0.083$. Two crystallographically different Eu(Pic)$_3$(TTD) units, in which the coordination numbers of two central ions (Eu1 and Eu2) are nine and ten, respectively, are present in the asymmetric unit. In the $^7F_0 \rightarrow {}^5D_0$ excitation spectrum of the complex, there are two sharp peaks at 578.5 and 579.0 nm indicating that the complex has two Eu(III) sites. Two different patterns in the $^5D_0 \rightarrow {}^7F_j$ (j = 1–4) luminescence spectrum have been observed (selectively excited $^5D_0$), which further reveals the existence of two Eu(III) sites compatible with the result of the structure determination.

Keywords: Europium picrate; TTD; Crystal structure; Luminescence spectra

INTRODUCTION

The development of luminescent chemical probes and sensors is the subject of intensive research, both in molecular materials and medical science. Probes based on Eu(III) are of special interest [1,2] because of narrow emission bands and spectroscopic properties in the visible region, and have been widely used as luminescence probes for calcium ion binding

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sites. In studies of the coordination selectivity of non-cyclic polyethers with different lanthanide ions, we found that lanthanide picrate complexes with the ligand *N*,*N*,*N′*,*N′*-tetraphenyl-3,6,9-trioxaundecanediamide (TTD) have different structures with diverse coordination numbers. Light and middle lanthanides are both nine and ten-coordinated [3], while heavy lanthanide ions are only nine-coordinated [4]. These kinds of unique and changing structures are important in investigating structure rules for lanthanide complexes, understanding the energy transfer between the central lanthanide ions and ligands and exploring correlation between crystal structures and fluorescent properties [1,2]. In this paper, we present the synthesis, single-crystal structure and time-resolved luminescence data for the title complex, and discuss the relationship between its emission spectrum and structure.

EXPERIMENTAL

Reagents

Europium (III) picrate [3] and TTD [4] were prepared according to literature methods. All solvents used were purified by standard methods.

Eu(Pic)$_3$(TTD)

A solution of 0.1 mmol of TTD in 15 cm^3 of anhydrous ethanol was added dropwise to a solution of 0.1 mmol of europium(III) picrate in 20 cm^3 of anhydrous ethanol. The mixture was stirred at room temperature for 2 h. The precipitated solid complex was filtered, washed with anhydrous ethanol and dried under vacuum over P_4O_{10} for 48 h. The complex was recrystallized from MeCN by slow evaporation to obtain yellow crystals of [Eu(Pic)$_3$(TTD)].

X-ray Structure Determination and Spectroscopic Measurements

Crystal data: $C_{50}H_{38}N_{11}O_{26}Eu$; $Mr = 1360.9$, Orthorthombic, space group *Pcab*, $a = 23.714(4)$, $b = 29.245(6)$, $c = 32.035(8)$ Å, $V = 22216(7)$ Å^3, $Z = 16$, $Dc = 1.63$ g cm^{-3}, $F(000) = 10976$, μ (MoKα) = 12.5 cm^{-1}. Intensity data for a crystal 0.18 × 0.30 × 0.60 mm in size were measured at 21°C on a R3M/E four-circle diffractometer with graphite-monochromatized MoKα radiation using $\omega/2\theta$ scans, Lorentz and polarization corrections were applied but no absorption correction was made.

The structure was solved by the Patterson method and subsequent difference Fourier techniques, and refined by block-matrix least-squares procedures, based on F. Non-hydrogen atoms were refined anisotropically. The weighting scheme was $w=[\sigma^2(F)+0.00010F^2]^{-1}\{1-\exp[-5(\sin\theta/\lambda)^2]\}$, with final $R_w=0.069$, $R=0.083$.

All calculations were performed on an Eclipse/S 140 computer with the SHELXTL program. Final atomic coordinates are given in Table I. Table of anisotropic thermal parameters, hydrogen atom coordinates and structure factors are available from W.L. upon request.

Luminescence spectra were measured by a method described in literature [5]. Time-resolved luminescence spectra were using a QUANTEL 571c-10 YAG: Nd laser instrument. IR spectras were recorded on a Nicolet 170SX FT-IR instrument using KBr discs in the 4000–220 cm^{-1} region.

RESULTS AND DISCUSSION

Crystal Structure of the Europium Complex

The structure of [Eu(Pic)$_3$(TTD)] is shown in Fig. 1 and selected bonding parameters are given in Table II. In the asymmetric unit there are two independent [Eu(Pic)$_3$(TTD)] monomers designated [Eu1(Pic)$_3$(TTD)] and [Eu2(Pic)$_3$(TTD)], the structures of which are different. The Eu1 ion is 10-coordinated by five oxygen atoms of TTD and five oxygen atoms of one unidentate and two bidentate picrates, while Eu2 ion is 9-coordinated by five oxygen atoms of TTD and four oxygen atoms of one bidentate and two unidentate picrates. The coordination geometry of Eul is best described as a distorted bicapped tetragonal antiprism with C_2 symmetric and as a distorted monocapped tetragonal antiprism with C_{2v} arrangement for Eu2.

In the two dependent monomers, the five oxygen atoms of TTD are not quite coplanar, their shifts from the mean plane being in the range 0.0525–0.996 Å, for [Eu1(Pic)$_3$(TTD)] and 0.2241–0.185 Å for [Eu2(Pic)$_3$(TTD)]. Europium lies out of these plane by 0.236 Å (for Eu1) and 0.175 Å (for Eu2). In each of the [Eu(Pic)$_3$(TTD)] monomers, TTD acts as a pentadentate ligand to form a ring-like coordination structure together with one oxygen atom from the bidentate picrate at the open side of the TTD chain [3].

The average distance between coordinated oxygen and Eu is 2.524 for Eu1, and 2.485 Å for Eu2. The four types of average bond lengths are in the order Eu-O (C-O, Pic) < Eu-O (C = O, TTD) < Eu-O (NO_2, Pic). This suggests that the picrate ion has stronger coordination ability.

TABLE I Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$) for the complex. U_{eq} is defined as one-third of the trace of the orthogonalized U_{ij} tensor

Atom	*x/a*	*y/b*	*z/c*	U_{eq} (Å^2)	*Atom*	*x/a*	*y/b*	*z/c*	U_{eq} (Å^2)
Eu1	8059(1)	3127(1)	2515(1)	40(1)	C50	8160(7)	1785(4)	2558(5)	42(4)
Eu2	2187(1)	5203(1)	5227(1)	38(1)	N12	1149(5)	4457(4)	6182(4)	47(4)
N1	9505(5)	2495(5)	3275(4)	49(4)	N13	3757(5)	4959(4)	4430(4)	52(4)
N2	6472(6)	2674(4)	1786(4)	48(4)	N14	2199(7)	5505(4)	6498(4)	61(4)
N3	9764(6)	3272(5)	2044(5)	53(4)	N15	4007(8)	5087(6)	7109(6)	100(5)
N4	10030(9)	2960(7)	568(6)	132(5)	N16	3914(7)	5484(5)	5639(5)	75(4)
N5	8082(6)	3060(6)	1150(5)	112(5)	N17	2021(8)	5291(6)	3894(6)	98(5)
N6	6186(6)	3469(5)	2889(4)	67(4)	N18	591(10)	4238(6)	3410(5)	119(5)
N7	6079(6)	3197(5)	4396(4)	73(4)	N19	602(7)	4659(5)	4843(5)	79(4)
N8	7879(6)	3152(5)	3694(4)	61(4)	N20	1657(8)	3747(5)	4539(6)	118(5)
N9	7226(6)	1978(5)	3464(4)	70(4)	N21	2976(8)	2677(4)	5134(6)	102(5)
N10	8114(11)	603(6)	2936(8)	146(5)	N22	3063(6)	4267(4)	5583(5)	61(4)
N11	8444(6)	2008(4)	2209(4)	49(4)	O27	1821(4)	4804(4)	5813(3)	50(3)
O1	8882(4)	2666(3)	2780(3)	44(3)	O28	1259(5)	5500(3)	5628(4)	52(3)
O2	8945(4)	3515(3)	2828(3)	46(3)	O29	1816(5)	6079(4)	5151(4)	66(4)
O3	8158(4)	4039(3)	2499(4)	51(3)	O30	2738(4)	5772(3)	4757(4)	50(3)
O4	7342(4)	3617(3)	2058(3)	48(3)	O31	3004(5)	4960(4)	4834(4)	62(4)
O5	7295(5)	2769(4)	2122(4)	58(4)	O32	2725(4)	5577(3)	5719(3)	47(3)
O6	8594(4)	3301(3)	1939(3)	45(3)	O33	1947(6)	5230(4)	6731(4)	83(4)
O7	9894(6)	2945(5)	2258(4)	82(4)	O34	1973(5)	5834(4)	6346(4)	73(4)
O8	9823(6)	3663(5)	2161(4)	82(4)	O35	3734(8)	5045(6)	7446(6)	121(5)
O9	10501(9)	3075(7)	636(6)	140(5)	O36	4510(7)	5048(6)	7063(5)	132(5)
O10	9816(10)	2739(8)	301(9)	177(5)	O37	4285(8)	5227(7)	5545(6)	146(5)
O11	7927(8)	2826(6)	884(6)	133(5)	O38	3782(7)	5801(6)	5406(6)	130(5)
O12	7833(10)	3384(7)	1273(7)	169(5)	O39	1583(5)	5248(4)	4679(3)	57(3)
O13	7314(5)	3391(4)	2929(4)	64(4)	O40	2488(7)	5141(7)	3902(7)	150(5)
O14	6098(7)	3857(5)	2781(5)	94(4)	O41	1947(9)	5698(6)	3882(6)	145(5)
O15	6056(6)	3143(5)	2679(4)	93(4)	O42	854(10)	4205(6)	3078(6)	160(5)
O16	5588(6)	3286(5)	4370(5)	101(4)	O43	169(9)	4038(6)	3491(6)	160(5)
O17	6320(7)	3081(5)	4716(5)	112(4)	O44	38(8)	4484(7)	4855(6)	146(5)
O18	8101(6)	3095(5)	4031(4)	101(4)	O45	873(6)	4736(5)	5164(4)	97(4)
O19	8196(5)	3161(4)	3376(4)	74(4)	O46	2092(4)	4443(3)	5026(3)	51(3)
O20	7756(5)	2460(4)	2851(3)	48(3)	O47	1798(10)	3891(7)	4212(6)	167(5)
O21	6821(7)	2165(5)	3353(5)	115(4)	O48	1202(8)	3736(7)	4697(8)	170(5)
O22	7418(8)	2025(6)	3803(6)	132(5)	O49	2762(10)	2415(4)	4892(6)	169(5)
O23	7880(10)	392(8)	3212(7)	177(5)	O50	3362(8)	2598(5)	5366(5)	125(5)
O24	8402(9)	416(7)	2668(6)	156(5)	O51	3488(7)	4149(5)	5775(5)	104(4)
O25	8826(5)	1795(4)	2020(4)	70(4)	O52	2901(5)	4651(3)	5607(3)	53(3)
O26	8327(5)	2394(4)	2107(4)	55(4)	C51	1485(7)	4051(6)	6206(6)	52(4)
C1	9678(7)	1870(6)	2786(5)	60(4)	C52	1921(9)	4022(6)	6498(6)	64(4)
C2	9726(8)	1397(6)	2703(6)	71(5)	C53	2261(10)	3618(8)	6521(8)	97(5)
C3	9630(9)	1076(7)	3011(7)	76(5)	C54	2111(9)	3260(7)	6249(7)	76(5)
C4	9504(10)	1232(8)	3418(7)	88(5)	C55	1697(10)	3293(7)	5952(8)	85(5)
C5	9456(9)	1715(7)	3508(7)	79(5)	C56	1347(8)	3690(6)	5934(7)	77(5)
C6	9537(7)	2012(6)	3185(6)	49(4)	C57	605(8)	4467(6)	6389(6)	64(4)
C7	9818(7)	2648(5)	3645(5)	51(4)	C58	117(7)	4294(7)	6185(7)	79(5)
C8	9501(8)	2703(6)	4022(7)	69(5)	C59	−397(8)	4309(7)	6410(8)	101(5)
C9	9814(9)	2840(7)	4381(7)	75(5)	C60	−402(10)	4490(8)	6812(8)	105(5)
C10	10373(10)	2907(7)	4372(7)	85(5)	C61	84(9)	4647(9)	7027(7)	117(5)
C11	10674(9)	2865(7)	3994(7)	83(5)	C62	589(8)	4624(8)	6795(7)	97(5)

TABLE I (*Continued*)

Atom	*x/a*	*y/b*	*z/c*	U_{eq} (Å^2)	*Atom*	*x/a*	*y/b*	*z/c*	U_{eq} (Å^2)
C12	10398(9)	2723(6)	3616(7)	68(5)	C63	1347(7)	4812(6)	5981(5)	56(4)
C13	9217(6)	2790(6)	3048(5)	40(4)	C64	985(7)	5234(6)	5946(5)	51(4)
C14	9321(7)	3297(5)	3116(5)	52(4)	C65	1049(8)	5972(6)	5609(6)	63(5)
C15	9013(7)	4014(6)	2880(5)	48(4)	C66	1222(8)	6148(6)	5209(7)	73(5)
C16	8731(7)	4217(5)	2515(6)	55(4)	C67	2026(8)	6338(7)	4794(8)	89(5)
C17	7864(8)	4286(6)	2161(6)	68(4)	C68	2653(8)	6273(4)	4788(7)	73(4)
C18	7276(8)	4111(6)	2151(5)	60(4)	C69	3257(7)	5682(5)	4550(6)	61(4)
C19	6843(7)	3449(5)	1866(5)	49(4)	C70	3328(6)	5172(6)	4606(5)	57(4)
C20	6881(7)	2937(5)	1935(5)	36(4)	C71	4172(7)	5218(7)	4186(6)	57(4)
C21	6036(8)	2857(6)	1516(6)	61(4)	C72	4588(9)	5468(7)	4395(7)	79(5)
C22	6153(8)	2950(6)	1113(6)	63(5)	C73	4995(11)	5705(9)	4134(10)	108(5)
C23	5677(10)	3113(8)	848(7)	88(5)	C74	4972(9)	5663(8)	3713(8)	95(5)
C24	5142(9)	3144(7)	1025(7)	78(5)	C75	4565(10)	5411(7)	3518(8)	91(5)
C25	5059(8)	3064(7)	1428(7)	70(5)	C76	4136(8)	5169(7)	3741(6)	73(5)
C26	5503(8)	2917(6)	1697(6)	57(4)	C77	3835(8)	4473(6)	4458(5)	56(4)
C27	6421(6)	2194(6)	1886(6)	47(4)	C78	4338(9)	4322(7)	4654(7)	77(5)
C28	6359(7)	1865(5)	1566(6)	67(4)	C79	4430(8)	3845(6)	4709(7)	99(5)
C29	6292(9)	1403(5)	1683(7)	84(5)	C80	4022(8)	3548(7)	4550(7)	102(5)
C30	6272(10)	1283(8)	2107(8)	96(5)	C81	3506(10)	3696(5)	4331(7)	96(5)
C31	6352(8)	1613(6)	2415(7)	75(5)	C82	3441(10)	4174(5)	4309(5)	80(5)
C32	6432(9)	2081(7)	2310(7)	84(5)	C83	3029(7)	5495(5)	6029(5)	43(4)
C33	8904(8)	3196(5)	1624(5)	49(4)	C84	2808(8)	5419(5)	6447(6)	52(4)
C34	9497(7)	3186(6)	1647(5)	50(4)	C85	3099(9)	5283(6)	6790(6)	66(4)
C35	9873(8)	3117(7)	1317(5)	62(4)	C86	3676(8)	5226(7)	6748(6)	76(5)
C36	9632(7)	3017(7)	925(5)	58(4)	C87	3941(9)	5280(7)	6359(7)	73(5)
C37	9043(8)	2979(6)	871(6)	68(5)	C88	3620(8)	5421(5)	6035(6)	53(4)
C38	8699(5)	3102(5)	1218(5)	45(4)	C89	1359(8)	5003(6)	4384(7)	60(4)
C39	7052(7)	3333(5)	3289(5)	48(4)	C90	1549(8)	4984(6)	3970(7)	66(5)
C40	6466(7)	3384(5)	3294(4)	41(4)	C91	1314(9)	4756(7)	3656(6)	73(5)
C41	6109(8)	3344(6)	3634(5)	51(4)	C92	842(11)	4492(6)	3756(6)	93(5)
C42	6405(7)	3248(5)	4003(5)	51(4)	C93	596(8)	4474(7)	4130(6)	72(5)
C43	6984(8)	3172(5)	4026(5)	57(4)	C94	868(8)	4718(7)	4441(6)	58(4)
C44	7290(6)	3222(5)	3666(5)	49(4)	C95	2310(6)	4046(5)	5068(5)	39(4)
C45	7841(7)	2028(4)	2846(4)	31(4)	C96	2123(7)	3670(5)	4836(5)	64(4)
C46	7593(7)	1753(5)	3157(5)	49(4)	C97	2309(8)	3227(7)	4846(7)	80(5)
C47	7646(9)	1287(6)	3206(6)	71(5)	C98	2767(9)	3146(4)	5108(6)	77(5)
C48	7996(9)	1098(4)	2890(5)	83(5)	C99	3009(8)	3479(6)	5350(6)	62(4)
C49	8282(8)	1311(5)	2570(5)	72(4)	C100	2788(7)	3922(5)	5337(4)	38(4)

Luminescence Spectra

The high-resolution $^7F_0 \rightarrow {}^5D_0$ excitation spectrum is composed of two sharp peaks at 578.5 nm and 579.0 mm, a weak peak and a weak shoulder peak at 579.1 and 579.3 nm indicated by monitoring the luminescence of Eu(III) from 580.2–517.8 nm at 77 K. This indicates two Eu(III) sites coexist in the crystal [6], in agreement with the structure determination.

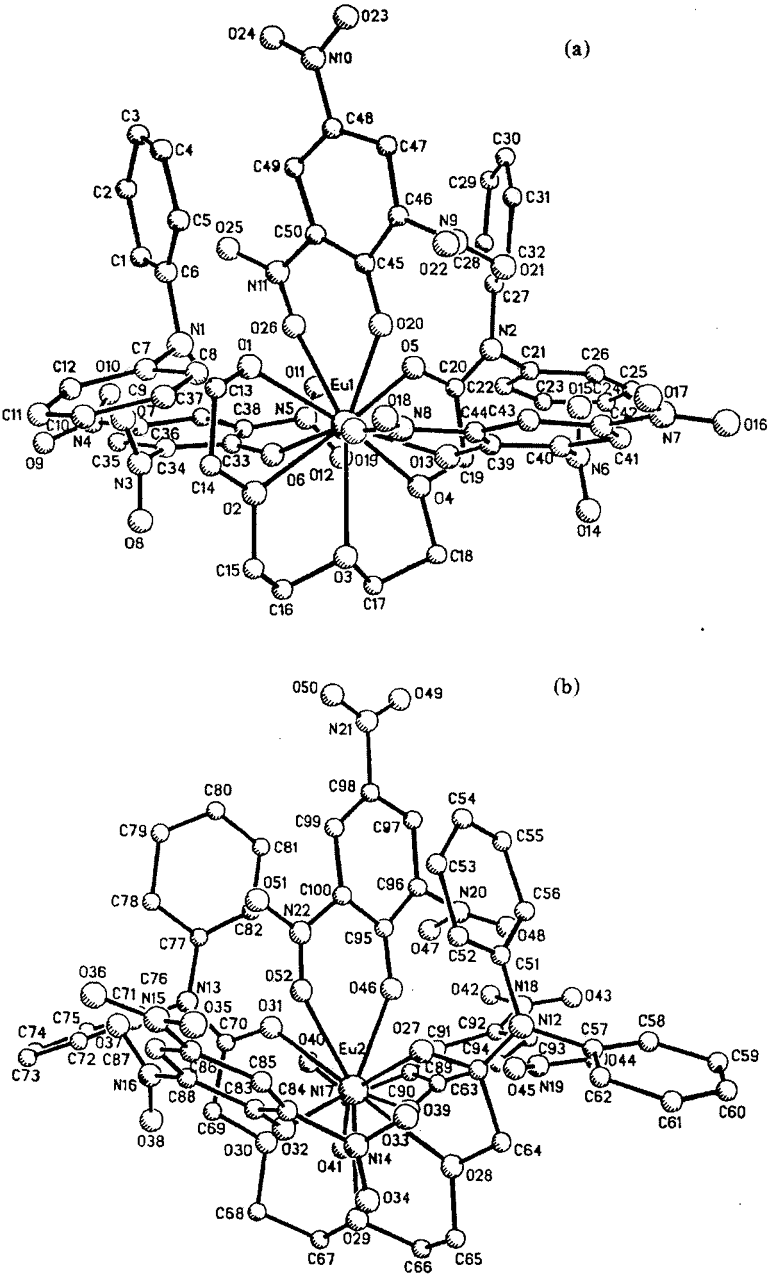

FIGURE 1 Molecular structures of [Eu1(Pic)$_3$TTD] (a) and [Eu2(Pic)$_3$TTD] (b), with the atom numbering scheme.

TABLE II Selected bond lengths (Å) and angles (deg) for the title complex

Eu(1)-O(1)	2.519(10)	Eu(2)-O(27)	2.375(10)
Eu(1)-O(2)	2.589(10)	Eu(2)-O(28)	2.693(11)
Eu(1)-O(3)	2.679(9)	Eu(2)-O(29)	2.20(11)
Eu(1)-O(4)	2.664(10)	Eu(2)-O(30)	2.97(10)
Eu(1)-O(5)	2.442(11)	Eu(2)-O(31)	2.16(11)
Eu(1)-O(6)	2.297(11)	Eu(2)-O(32)	2.303(10)
Eu(1)-O(13)	2.341(12)	Eu(2)-O(39)	2.269(11)
Eu(1)-O(19)	2.779(12)	Eu(2)-O(46)	2.325(9)
Eu(1)-O(20)	2.341(10)	Eu(2)-O(52)	2.638(10)
Eu(1)-O(26)	2.590(11)		
Mean Lengths			
Eu(1)-O(C=O, TTD)	2.480	Eu(2)-O(C=O, TTD)	2.395
Eu(1)-O(C-O-C, TTD)	2.664	Eu(2)-O(C-O-C, TTD)	2.670
Eu(1)-O(C-O, Pic-)	2.326	Eu(2)-O(C-O, Pic-)	2.299
Eu(1)-O(-NO_2, Pic-)	2.684	Eu(2)-O(-NO_2, Pic-)	2.638

To further research the correlation between structure and luminescence, we used 578.5, 579.0, 579.1 and 579.3 nm as analytical wavelengths, and the $^5D_0 \rightarrow ^7F_{1,2}$ transition emission spectra were recorded. The energy levels of the two Eu(III) sites were determined by corresponding IR and Raman spectra and results are listed in Table III. The excitation spectrum of site (I) is different from that of site (II), corresponding to the $^5D_0$ levels of Eu(III). This suggests that the Eu(III) ions in the complex have two different chemical environments, while a weak peak and a weak shoulder peak is seen in the selectively excited emission spectra. These are very similar and are assigned as vibronic peaks due to electron–phonon coupling [7]. The $^5D_0$ level energy for site I has higher energy, and there is much stronger luminescence from site II in the emission spectrum of site I. When site I was excited, site II with lower energy fluorescence is involved in energy transfer from site I. Such energy transfer is indicated by the time-resolved luminescence spectra of $^5D_0 \rightarrow ^7F_0$ transitions.

With the time-resolved luminescence spectra, the curve obtained with a decay time of 100 μs is very similar to that obtained by selectively exciting the complex with the $^5D_0$ energy for Eu2 (site II); a shoulder on the left-side of the main peak corresponds with the main peak position of site I. With extension of decay time, this shoulder disappears and the peak shape is identical with that of site II. This indicates that the energy has transferred from site I (higher energy) to site II (lower energy) completely and fluorescence arises from site II.

The crystal structure and luminescence spectra show the two sites in the title complex have striking differences. When the complex is selectively excited, energy transition between two Eu(III) sites shortens the lumines-

TABLE III Emission spectra details with assignments

E (cm^{-1})	ΔE (cm^{-1})	*IR* (cm^{-1})	*Raman* (cm^{-1})	*Assignment*
Site I				
17286	0			${}^5D_0 \rightarrow {}^7F_0$
17010	276			${}^5D_0 \rightarrow {}^7F_1$
16962	324			${}^5D_0 \rightarrow {}^7F_1$
16748	538			${}^5D_0 \rightarrow {}^7F_1$
16354	932			${}^5D_0 \rightarrow {}^7F_2$
16322	964			${}^5D_0 \rightarrow {}^7F_2$
16263	1023			${}^5D_0 \rightarrow {}^7F_2$
16167	1119			${}^5D_0 \rightarrow {}^7F_2$
16066	1220			${}^5D_0 \rightarrow {}^7F_2$
Site II				
17267	0			${}^5D_0 \rightarrow {}^7F_0$
16983	284			${}^5D_0 \rightarrow {}^7F_1$
16925	342			${}^5D_0 \rightarrow {}^7F_1$
16786	481			${}^5D_0 \rightarrow {}^7F_1$
16336	931	922		Vibr.
16325	942	941	943	Vibr.
16366	901			${}^5D_0 \rightarrow {}^7F_2$
16271	996			${}^5D_0 \rightarrow {}^7F_2$
16254	1013			${}^5D_0 \rightarrow {}^7F_2$
16082	1185	1170	1170	Vibr.
16058	1209			${}^5D_0 \rightarrow {}^7F_2$

cence lifetime. The interaction of excitation states between the two sites creates a changing chemical environment in the complex, so it may be possible to use the title complex as a luminescence probe in clinical medicine and in chemical structure analysis.

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