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Synthesis and Optical Properties of a Heterocarboxylate Complex with the Zn_2Eu Metal Core

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Abstract—It was shown that refluxing of europium(III) acetate hydrate with trifluoroacetic acid in a dioxane–acetonitrile mixture gives the polymer $\{[Eu(\mu\text{-}OOCCF_3)_3(OH_2)_2]\}_n$ (**I**) containing dioxane solvate molecules. The reaction of **I** with $[phen(\mu\text{-}OOC^tBu)_2(OOC^tBu)_2]$ (**II**) (phen = 1,10-phenanthroline) in CH_2Cl_2 at room temperature gives a precipitate, the recrystallization of which from acetonitrile affords an unusual trinuclear heterometallic heteroanionic $phen_2Zn_2Eu(\mu^3\text{-}OH)(OOC^tBu)_4(OOCCF_3)_2$ complex (**III**). The structure of the products was established from X-ray diffraction data (CCDC no. 2235937–2235939). The optical properties of complex **III** were studied.

Keywords: trifluoroacetates, heterometallic complexes, europium, luminescence, phosphorescence, crystal structure

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INTRODUCTION

Lanthanide complexes attract attention of researchers, first of all, owing to their unique luminescent properties [1, 2]. However, it may be difficult to attain an efficient photoexcitation of lanthanide ions because of the forbidden *f–f* transitions; this results in low extinction coefficients [3–5]. One way to enhance luminescence is to prepare heterometallic *d–f* complexes in which the transition metal/ligand (d block) can act as an energy donor for sensitization of lanthanide ion luminescence [6–10]. Therefore, the development of facile methods for the synthesis of heterometallic complexes containing both 3*d* and 4*f* metal ions attracts increasing attention [11–18].

Lanthanide compounds containing anions of strong acids, the displacement of which to the outer sphere results in the appearance of vacant orbitals, can be used as potential building blocks for the preparation of intricate heterometallic complexes and clusters via the reactions with bases. The metal-containing phen/DipyM$(OOCR)_2$ molecules (M = Zn, Co, R = Ph, Me, ^{t}Bu, phen = 1,10-phenanthroline, Dipy = 2,2'-dipyridyl), the basicity of which is determined by the nature of the substituents R in the carboxylate anion, can be used as the donor bases. Lanthanide trifluoroacetates are well-known; however, their main drawback as the starting compounds for these reactions is the presence of coordinated water molecules [19–26].

The purpose of the present study is to prepare europium trifluoroacetates in the presence of dioxane and acetonitrile donor molecules under drastic conditions (refluxing for 5 h), to use them for the preparation of a heterometallic complex with the Zn_2Eu metal core, and to study the optical properties of the resulting heterometallic complex.

EXPERIMENTAL

The complexes were synthesized in air using solvents without additional purification: acetonitrile (special-purity grade, Khimmed), dioxane (analytical grade, Khimmed), dichloromethane (reagent grade, Khimmed). The synthesis was performed using commercially available chemicals: europium acetate hydrate (Acros) and 1,10-phenanthroline (99%, Acros); $[Zn(OOC^tBu)_2]_n$ was prepared by a known procedure [27].

Elemental analysis was performed on a Carlo Erba EA 1108 automatic C,H,N-analyzer. IR spectra were recorded on a Perkin-Elmer Spectrum 65 FTIR spectrophotometer using the attenuated total reflectance (ATR) method in the 400–4000 cm^{-1} frequency range.

[†] Deceased.

The absorption spectra of solutions of the complex and a related salt in acetonitrile were recorded on a Jasco V-770 instrument in quartz cells with an 1-cm thick absorbing layer.

The luminescence and phosphorescence spectra of the samples were recorded on a Perkin-Elmer SL-45 spectrofluorimeter equipped with a fiber optic attachment for solid samples. The solid samples were placed into quartz cells of 6 mm in diameter. A pulsed xenon lamp was used as the excitation source. The phosphorescence spectra were recorded using a 100 μs delay after the pulse, and after that, the data were integrated for 1 ms.

Synthesis of $\{[Eu(\mu\text{-}OOCCF_3)_3(OH_2)_2]\cdot O_2C_4H_8\}_n$ (I). An excess of trifluoroacetic acid (0.5 mL, 6 mmol) was added to a suspension of $Eu(OOCMe)_3\cdot H_2O$ (0.2 g, 0.5 mmol) in an acetonitrile–dioxane mixture (1 : 1) (10 mL), and the mixture was refluxed for 5 h. The resulting solution was concentrated to 3 mL in an argon flow and slowly cooled to room temperature. The resulting large colorless crystals were separated from the mother liquor by decantation, washed with hexane, and dried in an argon flow. The yield of **I** was 0.25 g (83%).

For $C_{10}H_{12}O_{10}F_9Eu$		
Anal. calcd., %	C, 19.52	H, 1.97
Found, %	C, 19.04	H, 1.93

IR (KBr; ν, cm^{-1}): 3409 s.br, 2945 m, 2906 w, 2880 w, 1712 s, 1651 s, 1658 s, 1479 s, 1454 m, 1379 w, 1262 m, 1209 s, 1149 s, 1115 m, 1081 m, 1043 w, 897 w, 870 s, 804 w, 796 s, 725 s, 607 m, 521 m, 455 w.

Synthesis of $phen_2Zn_2(\mu\text{-}OOC^tBu)_2(OOC^tBu)_2]$ (II). Phen (0.03 g, 0.13 mmol) was added to a suspension of the complex $[Zn(OOC^tBu)_2]_n$ (0.1 g, 0.13 mmol) in dichloromethane (5 mL). The obtained solution was kept at room temperature for 24 h. The resulting transparent crystals were separated from the mother liquor by decantation, washed with hexane, and dried in an argon flow. The yield of **II** was 0.06 g (50%).

IR (KBr; ν, cm^{-1}): 3265 m, 2965 m, 2955 s, 2933 w, 2865 w,1601 m, 1558 s, 1485 s, 1433 s, 1404 m, 1353 m, 1313 w, 1221 s, 1155 m, 1032 m, 890 m, 778 s, 734 w, 615 w, 471 w, 416 w.

For $C_{44}H_{52}N_4O_8Zn_2$			
Anal. calcd., %	C, 59.00	H, 5.85	N, 6.25
Found, %	C, 58.04	H, 4.83	N, 13.50

Synthesis of $[phen_2Zn_2Eu(\mu\text{-}OOC^tBu)_4(\mu^3\text{-}OH)\text{-}(OOCCF_3)_2]$ (III). A solution of $[phen_2Zn_2\text{-}(OOC^tBu)_4]$ (0.1 g, 0.11 mmol) and complex **I** (0.055 g, 0.11 mmol) in dichloromethane (10 mL) was stirred at room temperature for 10 min. The white precipitate that formed was collected on a filter and dissolved in acetonitrile (10 mL). The resulting transparent solution was left for slow evaporation at room temperature for 24 h. The transparent crystals that precipitated were separated from the mother liquor by decantation, washed successively with cold benzene and hexane, and dried in an argon flow. The yield of **III** was 0.04 g (28%).

For $C_{48}H_{53}N_4O_{13}F_6Zn_2Eu$			
Anal. calcd., %	C, 44.67	H, 4.14	N, 4.34
Found, %	C, 45.11	H, 4.17	N, 3.89

IR (KBr; ν, cm^{-1}): 3438 w.br, 2963 w, 1737 s, 1719 s, 1679 s, 1626 w, 1588 w, 1520 w, 1486 w, 1437 m, 1428 m, 1349 w, 1321 w, 1224 m, 1182 s, 1207 s, 1197 c, 1144 s, 1135 s, 1051 w, 902 w, 869 w, 846 m, 835 m, 795 s, 773 m, 722 s, 728 s, 610 w, 521 w, 481 w.

Single-crystal X-ray diffraction study of **I**–**III** was carried out on a Bruker Apex II diffractometer equipped with a CCD detector (MoK_α, $\lambda = 0.71073$ Å, graphite monochromator) [28]. The semiempirical absorption correction was applied by the SADABS program [29]. The structure was solved by direct methods and refined by the least-squares method, first in the isotropic and then in the anisotropic approximation on F^2_{hkl}. The hydrogen atom positions were calculated geometrically and refined in the isotropic approximation by the riding model. All calculations were carried out using the SHELXL-2018/3 program package [30] and the Olex2 program [31]. In the structure of **II**, the *tert*-butyl groups are disordered over two positions with 0.827/0.173 occupancy ratio. In **III**, the *tert*-butyl groups are disordered over two positions with 0.73/0.27 and 0.62/0.38 occupancy ratios. The geometry of the metal atom polyhedra was determined using the SHAPE 2.1 program [32]. The crystallographic parameters and structure refinement details for **I**–**III** are summarized in Table 1. Selected bond lengths are given in Table 2.

The full set of X-ray diffraction data of the compounds was deposited with the Cambridge Crystallographic Data Centre (CCDC no. 2235937 (**I**), 2235938 (**II**), 2235939 (**III**)) and is available at deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif.

RESULTS AND DISCUSSION

The simplest lanthanide complexes with trifluoroacetate anions have been studied rather comprehensively. They are binuclear, $[M_2(\mu\text{-}OOCCF_3)_4\text{-}(OOCCF_3)_2(OH_2)_x]$, and contain different numbers of

Table 1. Main crystallographic data and X-ray experiment and structure refinement details for **I–III**

Parameter	Value		
	I	**II**	**III**
Molecular formula	$C_{10}H_{12}O_{10}F_9Eu$	$C_{44}H_{52}N_4O_8Zn_2$	$C_{48}H_{53}N_4O_{13}F_6Zn_2Eu$
Molecular weight	615.16	895.63	1290.64
System	Triclinic	Orthorhombic	Monoclinic
Space group	$P\bar{1}$	$Pccn$	$P2_1/c$
a, Å	9.742(2)	9.91(8)	14.141(7)
b, Å	10.200(2)	14.034(9)	20.762(10)
c, Å	11.206(4)	30.750(11	20.359(10)
α, deg	104.146(4)	90	90
β, deg	93.126(3)	90	90.488(7)
γ, deg	117.504(2)	90	90
V, Å^3	939.5(4)	4275(36)	5977(5)
Z	2	4	4
ρ(calcd.), g/cm^3	2.175	1.392	1.434
μ, cm^{-1}	3.472	1.179	1.908
$2\theta_{max}$, deg	4.726–52.000	5.206–51.988	4.002–48.870
F(000)	592	1872.0	2600.0
Number of reflections	8171	23762	45145
Number of unique reflections	3641	4208	9853
R_{int}	0.0313	0.1793	0.1794
Number of refined parameters	3464	266	629
GOOF	1.025	0.739	1.016
R_1, wR_2 ($I > 2\sigma(I)$)	$R_1 = 0.0293$, $wR_2 = 0.0730$	$R_1 = 0.0437$, $wR_2 = 0.0743$	$R_1 = 0.0767$, $wR_2 = 0.1854$
$\Delta\rho_{max}/\rho_{min}$, e/Å^3	1.14/–1.18	0.31/–0.48	0.84/–0.91

Table 2. Selected bond lengths (Å) for compounds **I–III**

Bond length, Å	**I**	**II**	**III**
Zn–O		1.918(3)–2.081(17)	1.988(8)–1.994(9)
Zn–N		2.107(3)–2.202(15)	2.094(11), 2.236(14)
Eu–O(H_2O; OH)	2.299(3)–2.430(3)		2.453(7)
Eu– O(OOCR)	2.375(3)–2.555(3)		2.348(11)–2.483(7)
Eu...Eu	4.861(11), 5.031(10)		
Eu...Zn			3.647(2), 3.694(2)
Zn...Zn		4.077(6)	3.500(10)

water molecules depending on the nature of the lanthanide.

It was suggested that the use of coordinating dioxane solvent for the synthesis of these compounds would decrease the number of water molecules in the complex on long-term refluxing [33, 34]. As a result, the reaction of trifluoroacetic acid with europium acetate hydrate in acetonitrile followed by the addition of dioxane and refluxing for 5 h gave rise to the polymer $\{[Eu(OOCCF_3)_3(OH_2)_2]\cdot O_2C_4H_8\}_n$ (**I**).

Complex **I** crystallizes in the triclinic space group $P\bar{1}$ with an inversion center between two europium

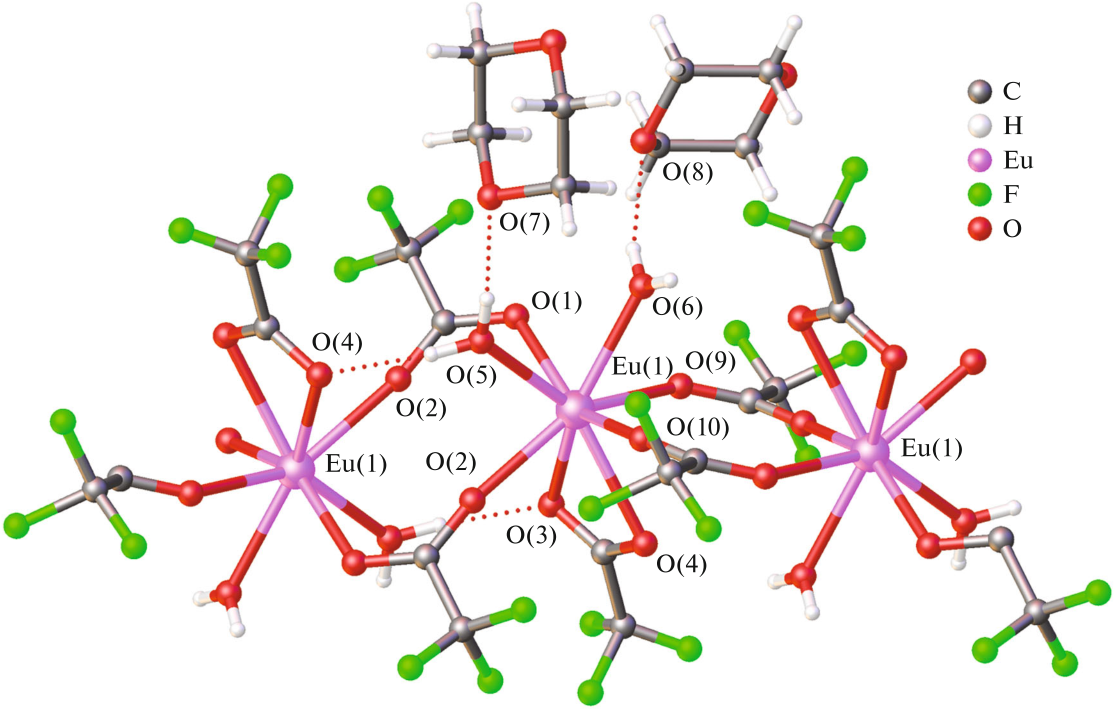

Fig. 1. Fragment of the polymer chain of **I**. The dashed line shows hydrogen bonds.

ions. In the structure of **I**, it is possible to distinguish binuclear $\{Eu_2\}$ groups with the metal–metal distance of 4.8605(11) Å, in which the metal ions are linked by two trifluoroacetate bridges (Eu–O, 2.354(3), 2.389(2) Å). The linear polymer chain directed along the *a* axis is formed via linking of the binuclear moieties by two $OOCCF_3$ bridges (Fig. 1; Eu–O, 2.399(3), 2.375(4) Å; Eu...Eu, 5.0131(10) Å; the EuEuEu angle is 160.00(1)°). Each europium ion additionally coordinates a chelating trifluoroacetate anion (Eu–O, 2.524(3), 2.555(3) Å) and two water molecules (Eu–O, 2.373(3), 2.430(3) Å), thus completing a square antiprism environment (S_q(Eu)) = 1.856). The coordinated water molecules are involved in the formation of a system of hydrogen bonds with the oxygen atoms of dioxane solvate molecules and coordinated trifluoroacetate anions, thus stabilizing the supramolecular layer (Table 3).

For obtaining a heterometallic compound by the reaction of zinc pivalate with 1,10-phenanthroline in dichloromethane, the complex $[phen_2Zn_2(\mu\text{-}OOC^tBu)_2(OOC^tBu)_2]$ (**II**, Fig. 2) was synthesized. In binuclear complex **II**, zinc ions are linked by two bridging pivalate anions (Zn–O, 1.982(3), 2.082(16) Å), being located at a nonbonding distance of 4.077(6) Å. The environment of each zinc atom is completed to a distorted square pyramid (S_q(Zn)) = 2.753) via monodentate coordination of a OOC^tBu anion (Zn–O 1.918(3) Å) and chelating phen molecule (Zn–N, 2.107(3), 2.201(15) Å). The coordinated phen molecules are involved in intra- and intermolecular π–π interactions to give supramolecular chains directed along the *b* axis (Table 3). The C–O...π and

Table 3. π–π Contacts in the crystal packing of complexes **II** and **III***

Contact	Cg···Cg, Å	Symmetry code	Cg···Pp, Å	α, deg
		II		
phen···phen	3.70(3)		3.4918(16)	6.61(19)
phen···phen	3.62(3)	5/2 – *x*, 1/2 – *y*, *z*	3.3954(19)	6.8(2)
		III		
phen···phen	3.570(9)	1 – *x*, 2 – *y*, –*z*	3.496(6)	1.0(7)
phen···phen	3.567(10)		3.479(7)	3.3(8)

* Cg is the aromatic ring centroid, Pp is the perpendicular to the ring plane, α is the angle between the planes of aromatic moieties.

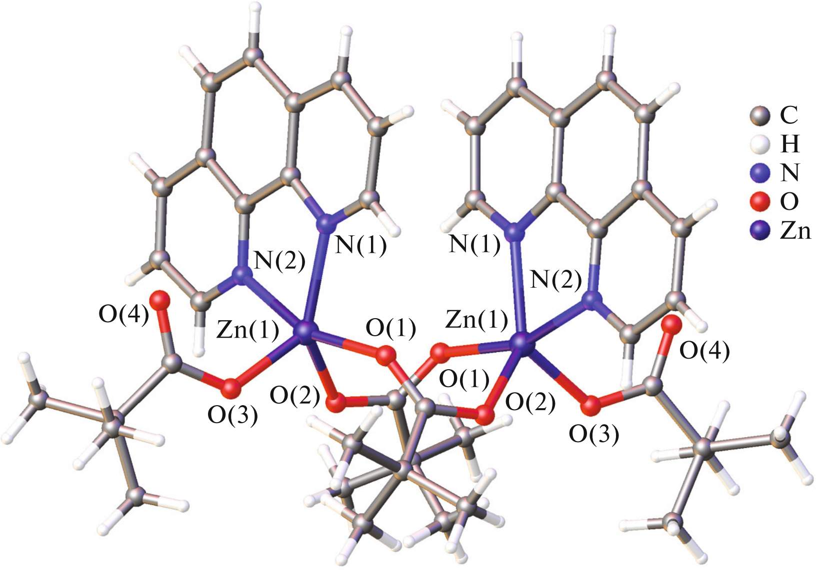

Fig. 2. Structure of complex **II**.

C–H...O contacts also participate in the crystal packing stabilization. Binuclear zinc complexes structurally similar to **II** have been obtained previously with 5-fluoro-2-hydroxybenzoate [35] and 3,5-dinitrobenzoate [36] anions. Zinc pivalate complexes with chelating ligands tend to exist as mononuclear structures [14, 17].

It was found that the reaction of two moles of compound **II** with one mole of **I** in acetonitrile at room temperature affords the heterometallic complex $[phen_2Zn_2(\mu^3\text{-}OH)(OOC^tBu)_4Eu(OOCCF_3)_2]$ (**III**, Fig. 3, 28% yield). According to X-ray diffraction data, in complex **III**, two phenZn moieties (Zn–N, 2.085(8)–2.223(10) Å; Zn...Zn, 3.4995(10) Å) are linked to the $Eu(\eta^2\text{-}OOCCF_3)(OOCCF_3)$ moiety (Eu–O, 2.362(8)–2.441(8) Å) via tridentate bridging hydroxyl group (Zn–O, 1.993(6), 1.999(6); Eu–O, 2.457(5) Å), three bridging pivalate anions (Zn–O, 1.986(6)–2.097(8); Eu–O, 2.372(7)–2.412(7)), and one bridging $OOCCF_3$ anion (Zn...Eu, 3.694(2), 3.647(2) Å; Zn–O, 2.005(9); Eu–O, 2.411(7) Å). Probably, the tridentate bridging hydroxyl group is formed in this reaction upon deprotonation of the water molecule coordinated to europium(III) in the starting trifluoroacetate giving weak pivalic acid, which results in relatively short Eu–Zn distances in the trinuclear complex **III**. The $\{Zn_2EuO\}$ group in complex **III** is non-planar, with the oxygen atom of the tridentate bridging hydroxyl group deviating from the Zn_2Eu plane by 0.478(8) Å.

The coordination environment of zinc corresponds to a trigonal bipyramid (S_q(Zn(1) = 1.705, S_q(Zn(2) = 0.627). Each europium ion completes the environment to a dodecahedron with triangular faces (S_q(Eu)) = 1.323) via coordination of the chelating OOC^tBu anion (Zn–O, 2.380(11), 2.440(12) Å). The coordinated bridging hydroxyl group is involved in hydrogen-bonding with the oxygen atom of the monodentately bound $OOCCF_3$ anion (Table 3). The Zn-coordinated phen molecules participate in the

Table 4. Parameters of hydrogen bonds in the crystals of complexes **I** and **III**

D–H···A	Distance, Å			DHA angle, deg	Symmetry code
	D–H	H...A	D...A		
			I		
O(5)–H(5A)···O(3)	0.85	2.26	120	2.788(5)	$-x, -y, 1-z$
O(5)–H(5B)···S(8)	0.85	1.94	153	2.728(5)	
O(6)–H(6A)···O(7)	0.86	1.85	166	2.684(5)	
O(6)–H(6B)···O(4)	0.86	1.97	157	2.780(5)	$1-x, -y, 1-z$
			III		
O(1)–H(1)···O(11)	0.98	1.89	151	2.786(13)	

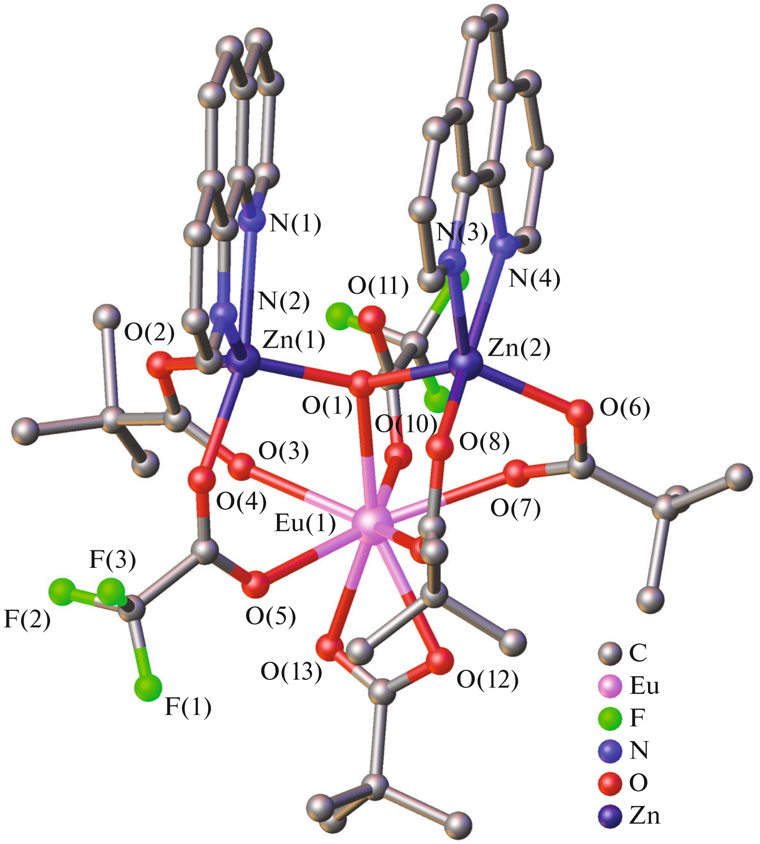

Fig. 3. Structure of complex **III**. The hydrogen atoms are omitted.

intra- and intermolecular π–π interactions to form supramolecular chains directed along the *b* axis (Table 4).

The optical properties of complex **III** were studied. The absorption spectrum of **III** is shown in Fig. 4. The line structure characteristic of phenanthroline is clearly seen in the spectrum (peaks at 230 and 270 nm, respectively). The extinction coefficient is relatively high ($\varepsilon = 10^5$ L mol^{-1} cm^{-1}).

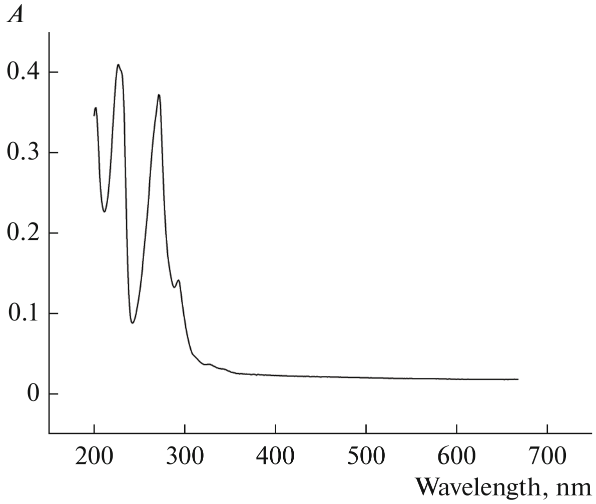

Fig. 4. Absorption spectra of complex **III**, $c = 4 \times 10^{-6}$ mol/L in acetonitrile.

The luminescence and excitation spectra of complex **III** are shown in Fig. 5. The long-wavelength shoulder in the emission spectrum in this case is due to intrinsic luminescence of phenanthroline [37]. The line at 617 nm corresponds to the $^5D_0 \rightarrow {}^7F_2$ transition in the Eu^{3+} ion; lines corresponding to other possible Eu^{3+} transitions have low intensity and are actually not manifested. The characteristic smooth pattern of the excitation spectrum in the 250–400 nm range attests to the presence of rather efficient energy transfer from organic ligands to the central europium ion.

The phosphorescence spectra of **III** (Fig. 6) are somewhat different from the luminescence spectra. The second phosphorescence line of the europium ion (593 nm) corresponding to the $^5D_0 \rightarrow {}^7F_1$ transition is clearly seen in the spectra; however, its intensity is much lower than that of the principal line at 617 nm. The unsymmetrical highly distorted environment of the europium ion in complex **III** affects the intensity ratio of the bands in the luminescence and phosphorescence spectra.

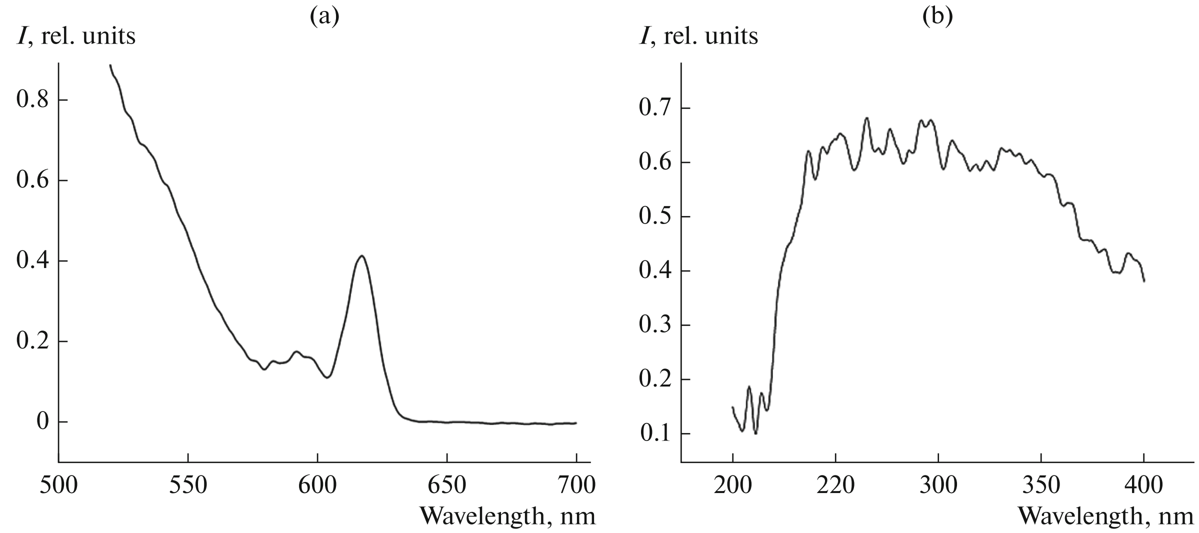

Fig. 5. (a) Luminescence spectrum at λ_{exc} = 375 nm and (b) excitation spectrum at λ_{emis} = 617 nm for complex **III**.

Thus, a procedure for the synthesis of unusual hydroxy-bridged mixed-anion complex with the Zn_2Eu metal core was developed. In the luminescence spectrum of the complex, the intrinsic luminescence of the ligand predominates, while europium luminescence lines have low intensity and are actually not manifested. The phosphorescence spectrum has two lines, but the principal phosphorescence line of the europium atom at 617 nm predominates.

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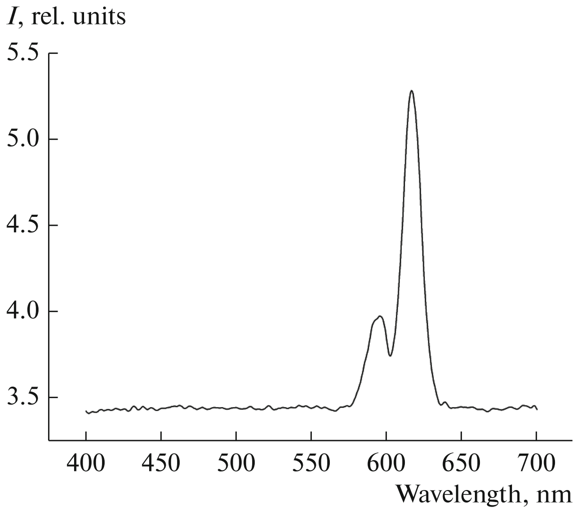

Fig. 6. Phosphorescence spectrum of complex **III**.

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CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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