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Preparation and Luminescent Properties of Coordination Polymers of Terbium and Europium with 1,4-Bis-(4-methoxycarbonyl-3-hydroxyphenoxycarbonyl)butane

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Abstract—Physicochemical properties of new isostructural homo- and heteronuclear coordination polymers of terbium and europium with 1,4-bis-(4-methoxycarbonyl-3-hydroxyphenoxycarbonyl)butane (H_2L) differing in the composition have been studied. The $Tb_2L_3·H_2O$ compound has exhibited efficient sensibilized green luminescence, whereas the corresponding europium-containing polymer $EuL_3·H_2O$ has shown practically no luminescence due to mismatch of energies of the ligand triplet level and the Eu^{3+} ion emitting level. Heteronuclear terbium-europium polymers ($Tb_{1.5}Eu_{0.5}L_3·H_2O$, $TbEuL_3·H_2O$, and $Tb_{0.5}Eu_{1.5}L_3·H_2O$) show luminescence via the intramolecular transfer of the exiting energy from Tb^{3+} emitting level to Eu^{3+}.

Keywords: terbium, europium, coordination polymer, luminescence

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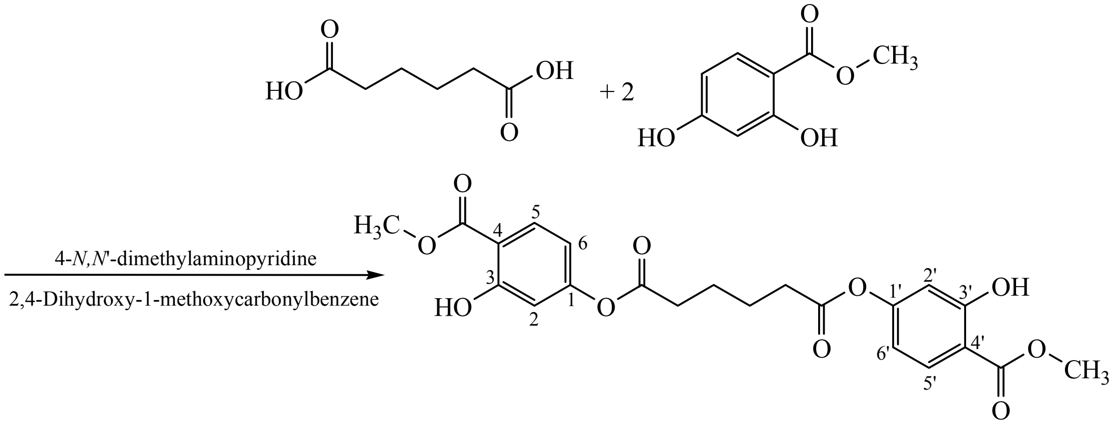
Aliphatic dicarboxylic acids and their derivatives has been widely used in coordination chemistry to prepare organometallic scaffold structures (metal-organic frameworks, MOF), that are in turn applied to produce sorbents, catalysts, and sensors [1–5]. Polymethylene chain of the dicarboxylic acid acts as a spacer for the coordination nodes of the complex to form a three-dimensional network. The MOF-type coordination polymers are usually prepared under hydrothermal synthesis conditions; single crystals suitable for X-ray diffraction analysis can be so obtained. Common drawbacks of the method are the poor applicability to industrial preparation and restricted possibility to control the synthesis process.

Several MOF-type complexes of lanthanide ions with dicarboxylic acids (including adipic acid) have been isolated and described [6–10]. Coordination compounds of lanthanides are known for the photoluminescence via the excitation energy transfer [11, 12]. In particular, weak luminescence of Tb(III) and Eu(III) ions was described in the above-cited papers; that was assigned to the low absorbance of the complex. The luminescence could be enhanced in the presence of the Lewis bases, in particular, 4,4'-bipyridine [13]. Besides using of the additional ligands, the luminescence could be strengthened by certain donor fragments introduced to the coordination node of the complex; this fact was attributed to the enhanced stability of the complex and, therefore, the higher efficiency of the excitation energy transfer.

In this work we aimed to prepare the MOF-type luminescent coordination polymers via the conventional precipitation method. Possible practical applications of the so obtained hybrid products are discussed elsewhere [14]. The ligand used to prepare the complexes, 1,4-bis-(4-methoxycarbonyl-3-hydroxyphenoxycarbonyl)butane (H_2L) was prepared via esterification of adipic acid with 2,4-dihydroxy-1-methoxycarbonylbenzene in the presence of dicyclohexylcarbodiimide and 4-*N,N*'-dimethylaminopyridine [15] (Scheme 1).

The ligand contained two spatially separated methoxycarbonyl and hydroxy groups, both capable of complex formation with metal ions [16–20]. The presence of such groups was shown to enhance terbium ions luminescence [21]. Phenylsalicylate was

Scheme 1.



demonstrated to form the structurally similar mixed complexes with terbium and europium; their crystalline precipitates exhibited sensibilized luminescence of europium(III) due to the transfer of excitation energy from terbium(III) ions [22]. In order to investigate such behavior in the case of H_2L acting as ligand we isolated its complexes with Tb^{3+} (**I**) and Eu^{3+} (**V**) as well as mixed complexes with the Tb : Eu ratio of 1.5 : 0.5 (**II**), 1 : 1 (**III**), and 0.5 : 1.5 (**IV**). According to the results reported in [23], the complex formation with methylsalicylate occurred in weakly basic medium (pH ≈ 9) and was accompanied with elimination of proton from the hydroxy group. Similarly, the complexes with H_2L could be formed after treating the reaction mixtures with ammonia solution. The complexes were insoluble in water and common organic solvents, including DMF and DMSO. The Tb(Eu) : H_2L ratio in the precipitates was 2 : 3, as determined by elemental analysis. Moreover, the complexes contained bound water. Thermograms and IR spectra of the isolated compounds were nearly identical, reflecting that the prepared complexes were likely isostructural.

According to thermal analysis data (Fig. 1) complex **I** lost 2.2% of the initial mass at 144–250°C, corresponding to a single water molecule with respect to a terbium(III) ion. The dehydration was not accompanied with any noticeable thermal effect. The onset of the complex decomposition (burning-out of the organic part) was observed at 265°C, as evidenced by a strong exothermic effect. The complex decomposition was complete at 700°C, the final product being Tb_4O_7.

IR absorption bands were assigned following the data reported elsewhere [24, 25]. In particular, the spectrum of the free ligand contained broadened band of CH_2 stretching vibrations of adipic acid fragment (maximum at 3188 cm^{-1}). In the spectrum of the complex this band was overlapped with a broad stretching vibrations band of hydrogen-bound water molecules. Symmetric and asymmetric stretching vibrations of OCH_3 groups were observed at 2955 and 2870 cm^{-1}, respectively.

H_2L molecule contained two ester groups in each of the terminal fragments. The ν(C=O) band of adipate ester was found at 1759 cm^{-1}. Carbonyl oxygen of that ester group was not involved in the complex formation; therefore, it was retained in the spectrum, although somewhat weakened (Fig. 2). The ν(C=O) vibration of methoxycarbonyl (methylsalicylate) group was assigned to the band at 1674 cm^{-1}. Similarly to the case of free methylsalicylate [25], it was shifted

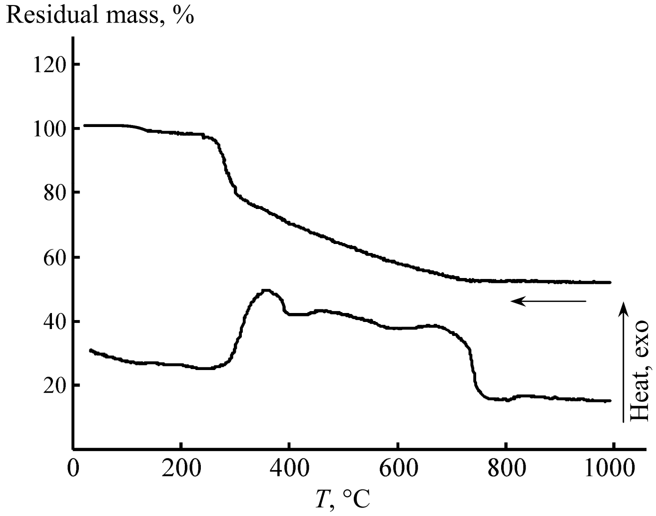


Fig. 1. Thermal analysis of the $Tb_2L_3·H_2O$ complex (**I**).

Table 1. Features of IR spectra of the H_2L ligand and its complex with terbium

H_2L	$Tb_2L_3{\cdot}H_2O$	Assignment
	3360 br	ν(OH) (H_2O)
3188 m. br	–	ν(CH_2) (adipate)
2955 m	2955 m	$\nu_{as}(CH_3)$
2870 w	2870 w	$\nu_s(CH_3)$
1759 vs	1759 s	ν(C=O) (adipate)
1674 vs	1655 vs. br	ν(C=O) (methylsalicylate)
1616 m		ν(Ph)
1585 m		ν(Ph)
1496 s	1495 m	ν(Ph)
1446 vs	1443 vs	ν(Ph)
1300 m	≈1300 m	ν(Ph)
1254 vs	≈1255 s	ν(O=C–O) (methylsalicylate)
1157 m	1161 m	ρ(CH_3)
1092 s	1092 s	ν(O–CH_3)
860 m	860 w	δ(Ph)
780 vs	≈789 m	γ(Ph)
694 s	702 w	γ(Ph)
567 s	563 w	δ(Ph)

towards low frequency due to the intramolecular hydrogen bonding. In the complex IR spectrum that band was observed at 1655 cm^{-1}. The band of ν(O–CH_3) vibration of H_2L molecule and its complexes was observed at 1092 cm^{-1}, similarly to the cases of the free methylsalicylate and its complexes.

According to IR spectroscopy data, coordination of H_2L with the metal ions occurred via the carbonyl oxygen atom of methylsalicylate fragment. Spectral changes accompanying the formation of the other studied complexes were similar to the above-described ones, thus confirming the structural similarity of the prepared compounds.

Hence, a set of physical properties investigated in this work (low solubility and high thermal stability) and the IR spectral data led to the conclusion that the individual as well as the mixed complexes of terbium and europium with H_2L were coordination polymers of two- or three-dimensional structure, containing the metal ions at the branching points. The ligand molecules were coordinated with both terminal groups to form a scaffold (network). Water molecules were likely located in the inner coordination sphere of the complexes.

The absorption spectrum of the free ligand H_2L in acetonitrile (1×10^{-4} mol/L) at 298 K contained a band with the molar absorptivity of ε_{max} 1.2×10^4 L mol^{-1} cm^{-1} (at λ_{max} = 299 nm). Energy of the ligand singlet $^1\pi\pi^*$ level as determined from the absorption maximum location was E_s = 33445 cm^{-1}.

In order to determine the energy of the ligand triplet level ($^3\pi\pi^*$) we studied its complex with gadolinium exhibiting the ligand phosphorescence (a broad band with λ_{max} 411 nm) upon excitation at λ = 290 nm at 77 K. The triplet level energy E_T of the ligand was thus 24330 cm^{-1}, higher than that of both emitting levels: $^5D_4$ of Tb^{3+} (20500 cm^{-1}) and $^5D_0$ of Eu^{3+} (17300 cm^{-1}). Therefore, H_2L could act as a photosensibilizer of those ions [26].

Excitation and emission spectra of Tb^{3+} in the $Tb_2L_3{\cdot}H_2O$ complex at room temperature are shown in Fig. 3, curve *1*. The excitation spectrum recorded at maximum of emission of the *f–f* band (corresponding to the terbium $^5D_4 \rightarrow {}^7F_5$ spectral transition) contained a wide band at 250–400 nm with a maximum at about 360 nm, assigned to the singlet ππ* state. The absence of the *f–f* absorption bands of Tb^{3+} showed that the sensibilization of luminescence via the ligand excited state was more efficient that direct excitation of Tb^{3+}. The emission spectrum of the $Tb_2L_3{\cdot}H_2O$ complex at room temperature (Fig. 3, curve *2*) (λ_{ex} 355 nm) contained characteristic bands of Tb^{3+} with maxima at 490, 545, 585, and 620 nm assigned to deactivation of the $^5D_4$ excited state via transitions to the corresponding sublevels of the ground state ^7F_j (j = 6, 5, 4, 3) of Tb^{3+}. The strongest band with maximum at 545 nm was assigned to the $^5D_4 \rightarrow {}^7F_5$ transition. The luminescence of the ligand was not observed, confirming the efficient transfer of energy from the ligand to the metal ion in the studied complex.

An extremely weak band of the $^5D_0 \rightarrow {}^7F_2$ *f–f* transition of Eu^{3+} was observed in emission spectrum of the $Eu_2L_3{\cdot}H_2O$ complex (**V**) (Fig. 4). No significant sensibilization of Eu^{3+} in the $Eu_2L_3{\cdot}H_2O$ complex was likely due to a noticeable energy gap between the ligand triplet state and the emitting level $^5D_0$ of Eu^{3+}, $\Delta_E(^3\pi\pi^*{-}^5D_0)$ = 7030 cm^{-1} (in the case of the terbium

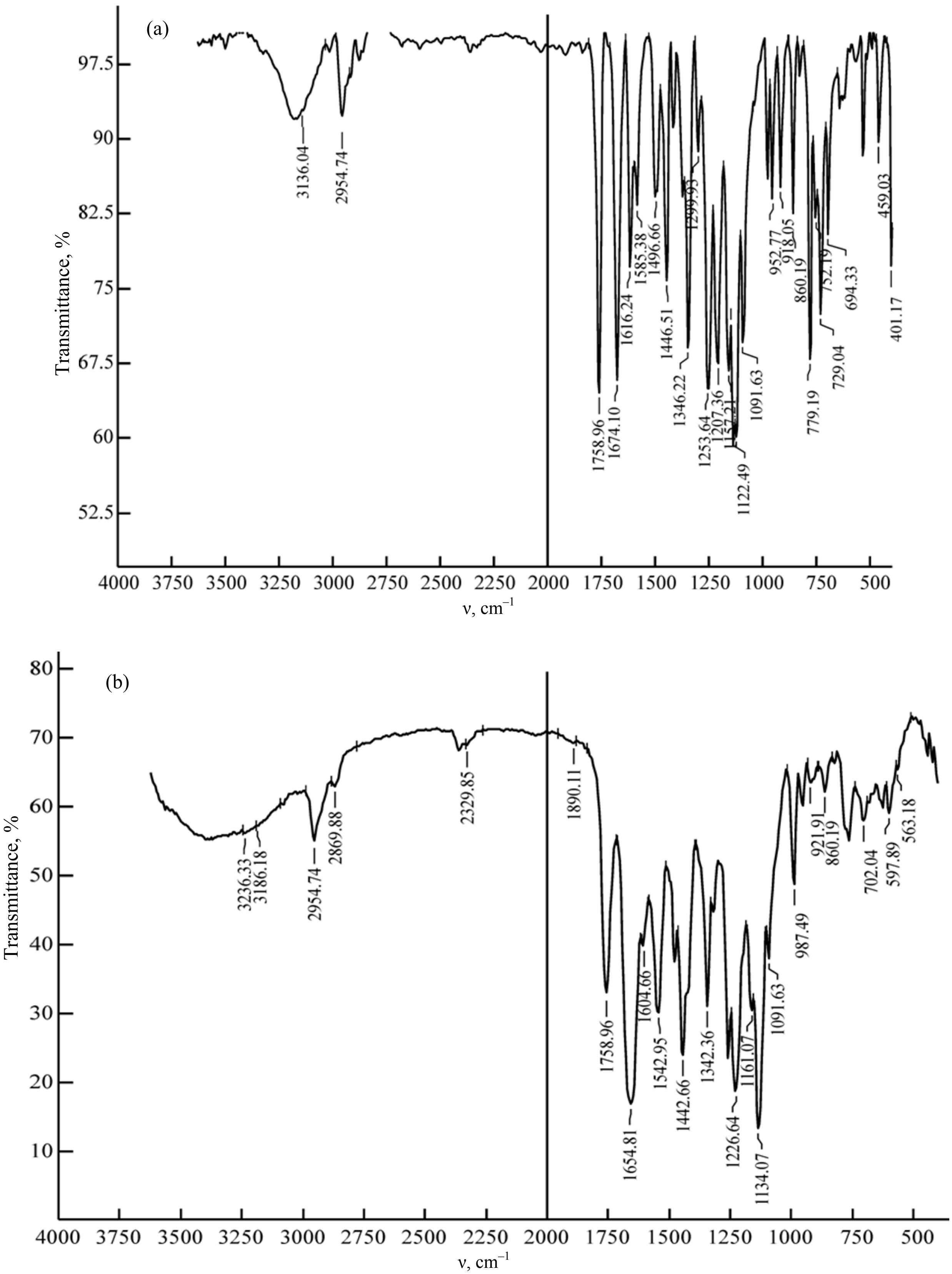


Fig. 2. IR spectra of the (a) H_2L ligand and (b) its complex with terbium.

complex **I** undergoing the photosensibilization the corresponding value was of Δ_E = 3830 cm^{-1}).

Emission spectra of the mixed terbium–europium complexes **II**–**IV** contained the *f*–*f* transition bands of both terbium and europium ions (Fig. 4). In the **II**–**III**–**IV** series the intensity of Eu^{3+} luminescence (λ = 615 nm, $^5D_0\rightarrow{}^7F_2$ transition) was enhanced, and the intensity of Tb^{3+} luminescence (λ = 545 nm, $^5D_4\rightarrow{}^7F_5$) decreased. Similar behavior was previously reported for polymeric mixed complexes [27] and mechanical mixtures of solid mononuclear complexes of terbium and europium [22, 28]; it was explained by transfer of excitation energy from emitting level of Tb(III) to that of Eu(III). The operation of the same mechanism in the case of the complexes studied in this work was confirmed by the measured lifetime of the excited

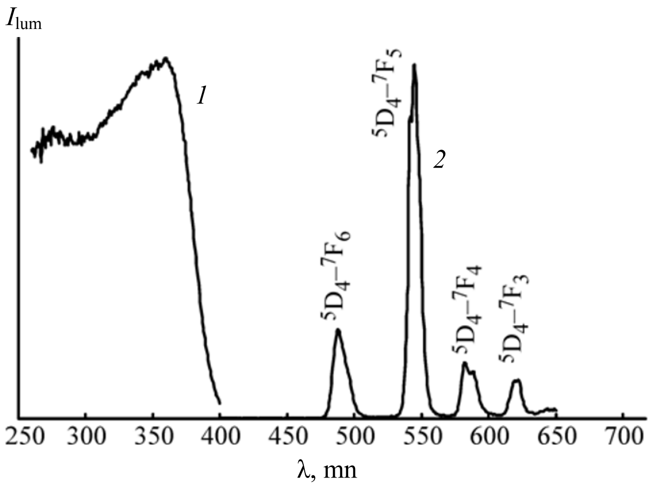


Fig. 3. Excitation (λ_{em} = 545 nm) (*1*) and emission (λ_{ex} = 355 nm) (*2*) spectra of the solid $Tb_2L_3{\cdot}H_2O$ complex.

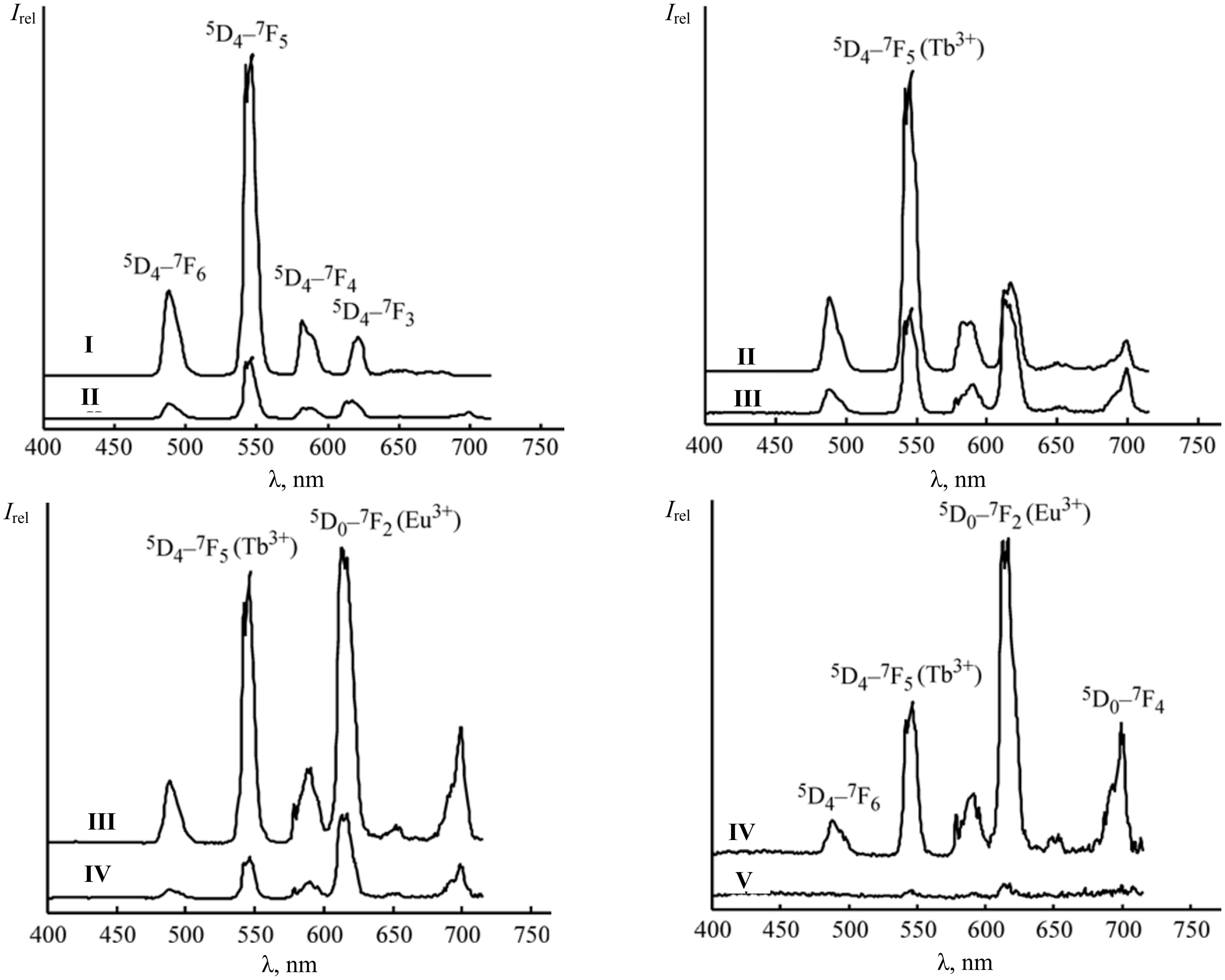


Fig. 4. Luminescence spectra (λ_{ex} 355 nm) of the $Tb_2L_3{\cdot}H_2O$ (**I**), $Tb_{1.5}Eu_{0.5}L_3{\cdot}H_2O$ (**II**), $TbEuL_3{\cdot}H_2O$ (**III**), $Tb_{0.5}Eu_{1.5}L_3{\cdot}H_2O$ (**IV**), and $Eu_2L_3{\cdot}H_2O$ (**V**) complexes (intensity scales are variable).

states (τ). From the data collected in Table 2 it follows that in the average lifetime of excited Tb^{3+} ion was lowered in the mixed complex **III** (0.700 ms) as compared with that in the homonuclear complex **I** (1.118 ms). At the same time, the lifetime of Eu^{3+} excited state in the complex **III** (0.342 ms) was longer than that in the homonuclear complex **V** (0.221 ms). The efficiency of the energy transfer (η) from terbium to europium could be estimated as follows in the case of purely dipole–dipole interactions, via the spatial transfer [29]:

$$\eta = 1 - \tau/\tau_0,$$

where τ and τ_0 standing for lifetimes of the donor (Tb^{3+}) excited state in the presence and in the absence of the acceptor (Eu^{3+}), respectively. The calculated values (50–70%, Table 2) confirmed the high efficiency of the energy transfer.

To conclude, in this work we prepared and studied new thermally stable coordination polymers of Tb(III) and Eu(III) based on 1,4-bis(4-methoxycarbonyl-3-hydroxyphenoxycarbonyl)butane. The complexes were prepared via precipitation route and were likely to be of the MOF-type structure. UV irradiation of the terbium complex produced green luminescence, whereas red-orange luminescence (typical of Eu^{3+} ion) was observed in the cases of the mixed complexes. The prepared compounds can be potentially applied in color displays fabrication or as luminescence sensors and structural probes.

EXPERIMENTAL

^1H NMR spectrum of the ligand solution in $CDCl_3$ was recorded using a Bruker AVANCE DRX 500 spectrometer (500 MHz, TMS as internal reference). Absorption spectra were recorded with a Perkin Elmer Lambda-9 UV/VIS/NIR spectrophotometer; emission spectra were registered using a SDL-1 spectrometer and Fluorolog 3-22 fluorimeter (Horriba Jobin Yvon). Luminescence was excited with a DRSh-250 mercury lamp of the SDL-1 spectrometer (λ = 254 and 365 nm radiation was filtered with UFS-1 and UFS-2 filters). Emission spectra of the complexes were recorded at 400–750 nm. Energy of triplet T_1 level of the ligand (E_{T1}, cm^{-1}) was determined from phosphorescence spectra of its complex with gadolinium(III) at 77 K. Lifetime of the excited state (τ) was measured using a Fluorolog 3-22 spectrofluorimeter. IR spectra were obtained with a FT-IR-8400 S spectrometer (Shimadzu) (400–4000 cm^{-1}, KBr).

Table 2. Average lifetime of terbium(III) and europium(III) ions in the complexes and the energy transfer efficiency

Compound	τ_{Tb}, ms	τ_{Eu}, ms	η, %
Tb_2L_3	1.813		
$Tb_{1.5}Eu_{0.5}L_3$	0.857	0.346	52.7
$TbEuL_3$	0.700	0.342	61.0
$Tb_{0.5}Eu_{1.5}L_3$	0.545	0.256	70.0
Eu_2L_3		0.221	

TGA curves were recorded in air using a Q-1000 derivatograph. The specimens with mass of about 50 mg were heated in an alumina crucible at a rate 10 deg/min (20–1000°C).

The metals content in the complexes was determined via titration with EDTA after combustion of the specimen and dissolution of the residue in perchloric acid.

1,4-Bis(4-methoxycarbonyl-3-hydroxyphenoxycarbonyl)butane (H_2L). 0.5 g (0.0043 mol) of 4-*N,N'*-dimethylaminopyridine was added at stirring to 7.0 g (0.043 mol) of 2,4-dihydroxy-1-methoxycarbonylbenzene and 3.0 g (0.2 mol) of adipic acid in anhydrous chloroform. The mixture was cooled to 0°C, and 8.3 g (0.04 mol) of dicyclohexylcarbodiimide was added. The reaction mixture was stirred at 0°C during 10 min, and then at room temperature during 3 h. The formed precipitate was filtered off and washed with anhydrous methylene chloride (2×15 mL). The solvent was evaporated under a reduced pressure, and the formed precipitate was washed with methylene chloride (30 mL) to remove residual urea. The product was washed with hot ethanol (2 × 30 mL). Yield 5 g (56%), mp 110°C. ^1H NMR spectrum, δ, ppm (*J*, Hz): 10.86 s (1H, OH), 7.83 d (2H, $H^{5,5'}$, J^3 8.72), 6.84–6.52 m (4H, $H^{2,6,2',6'}$), 3.93 s (6H, CH_3O), 2.61 br.s [4H, $C(O)CH_2$], br.s (4H, CH_2CH_2). Found, %: C 59.00; H 4.92. $C_{22}H_{24}O_{10}$. Calculated, %: C 59.19; H 4.96.

Due to low solubility of H_2L in most of common organic solvents, the complexes were prepared in THF or acetonitrile (the ligand was moderately soluble in those media at heating). Better reproducible results were obtained when using acetonitrile.

Monoaquatris[1,4-bis(4-methoxycarbonyl-3-hydroxyphenyloxycarbonyl)butano]diterbium $Tb_2L_3 \cdot H_2O$

(I). 268 mg (0.6 mmol) of H_2L was dissolved at slight heating in 30 mL of acetonitrile, and 4 mL of 0.1085 mol/L solution of $Tb(ClO_4)_3$ (0.434 mmol) was added. Then, aqueous ammonia (1 : 1) was added dropwise till the precipitate started forming. After keeping overnight the formed precipitate was filtered off, washed with acetonitrile, and dried. The complex was insoluble in water and common organic solvents, including DMF and DMSO. Yield 0.34 g (74.1%). Found, %: Tb 18.74; C 47.1; H 4.1. $Tb_2C_{68}H_{68}O_{31}$. Calculated, %: Tb 19.96; C 47.48; H 3.96.

Monoaquatris[1,4-bis(4-methoxycarbonyl-3-hydroxyphenyloxycarbonyl)butano]dieuropium $Eu_2L_3\cdot H_2O$ (V) was prepared similarly using equivalent amount of europium perchlorate instead of the terbium salt. Yield 0.38 g (74.7%). Found, %: Eu 18.25; C 48.00; H 4.02. $Eu_2C_{68}H_{68}O_{31}$. Calculated, %: Eu 18.38; C 47.88; H 3.99.

The mixed complexes were prepared similarly using both terbium and europium salts in the required ratios.

$Tb_{1.5}Eu_{0.5}L_3H_2O$ (II). Yield 0.30 g (72.1%). Found, %: Σ(TbEu) 19.15; C 47.24; H 4.04. $Tb_{1.5}Eu_{0.5}C_{68}H_{68}O_{31}$. Calculated, %: Σ(TbEu) 18.89; C 47.58; H 3.97.

$TbEuL_3H_2O$ (III). Yield 0.32 g (70%). Found, %: Σ(TbEu) 18.88; C 47.77; H 4.00. $TbEuC_{68}H_{68}O_{31}$. Calculated, %: Σ(TbEu) 18.72; C 47.68; H 3.97.

$Tb_{0.5}Eu_{1.5}L_3H_2O$ (IV). Yield 0.34 g (74.6%). Found, %: Σ(TbEu) 18.79; C 48.10; H 3.94. $Tb_{0.5}Eu_{0.5}C_{68}H_{68}O_{31}$. Calculated, %: Σ(TbEu) 18.55; C 47.78; H 3.98.

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