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Synthesis of Octafluorobiphenyl-4,4'-dicarboxylic Acid and Photoluminescent Compounds Based Thereon

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Abstract—Octafluorobiphenyl-4,4'-dicarboxylic acid (H_2L) and its complexes with Tb(III) and Eu(III) [$Ln_2(H_2O)_4(L)_3·3H_2O$ and $Ln_2(phen)_2(L)_3·2H_2O$] have been prepared; their structure has been elucidated from IR spectroscopy and X-ray diffraction analysis data. Thermal properties of the compounds have been studied. The complexes of Tb(III) and Eu(III) exhibit green and red photoluminescence, respectively.

Keywords: octafluorobiphenyl-4,4'-dicarboxylic acid, lanthanide, complex, photoluminescence

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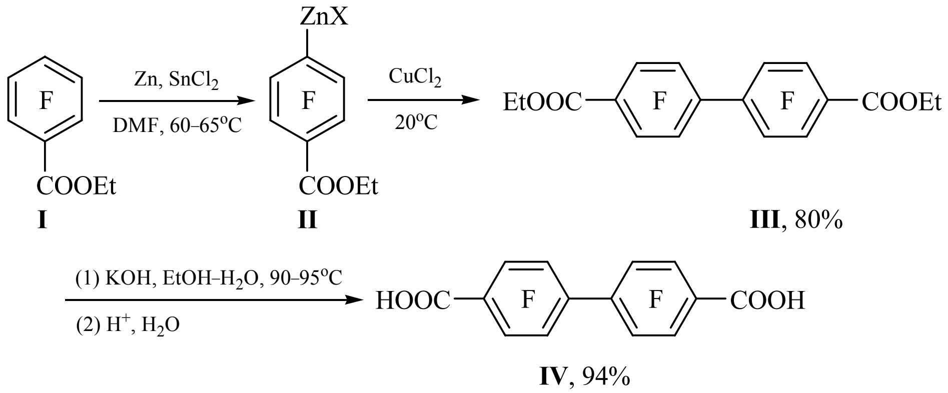
Luminescent coordination compounds of lanthanides (Ln) with ligands containing donor O and N atoms have been widely applied to the production of light emitting diodes and chemosensors [1–5]. The presence of F atoms in the ligand enhances the luminescence [2, 6, 7]; therefore, lanthanide complexes with aromatic perfluorinated carboxylic acids have been thoroughly investigated. In particular, photoluminescent compounds containing lanthanide cations and anions of C_6F_5COOH, 4-$CF_3C_6F_4COOH$, and 4-$(CF_3)_2CFC_6F_4COOH$ have been prepared [8–11]. Later, luminescent coordination polymers of lanthanides (erbium, terbium, gadolinium, europium, and lanthanum) with bridging anions of tetrafluoroterephthalic acid *p*-$HOOCC_6F_4COOH$ (H_2L') have been obtained [12, 13]. We have earlier synthesized the $[Tb_2(H_2O)_4(L')_3·2H_2O]_n$ and $Tb_2(phen)_2(L')_3·2H_2O$ coordination polymers [14] and other compounds: $Ln_2(H_2O)_4(L')_3·2H_2O$ and $Ln_2(phen)_2(L')_3·2H_2O$ (Ln = Eu, Sm, Dy; phen = phenanthroline) [15]. Synthesis of coordination compounds of Ce, Pr, Nd, Sm, Dy, Er, and Yb with tetrafluoroterephthalic and biphenyl-4,4'-dicarboxylic acids has been described in [16–18].

In this work we have developed a new procedure to obtain octafluorobiphenyl-4,4'-dicarboxylic acid (H_2L) and prepared its luminescent coordination compounds with Tb(III) and Eu(III).

Octafluorobiphenyl-4,4'-dicarboxylic acid was prepared as follows. First, the interaction of ethyl pentafluorobenzoate **I** with zinc in the presence of $SnCl_2$ in DMF afforded the corresponding (4-ethoxycarbonyl-2,3,5,6-tetrafluorophenyl)zinc compounds **II** [19] that were further transformed into diethyl octafluorobiphenyl-4,4'-dicarboxylate **III** under the action of anhydrous $CuCl_2$ [20]. Finally, alkaline hydrolysis of ester **III** afforded the target acid H_2L (**IV**) (Scheme 1).

Coordination compounds **V**–**VIII** were prepared via the interaction of $Ln(OH)_3$ (Ln = Tb or Eu) with the acid H_2L. Elemental analysis data of the prepared products are given in Table 1; the composition of compounds **V** and **VI** corresponded to the $Ln_2(H_2O)_4L_3·3H_2O$ formula. The results coincided with the thermal analysis data. The TG curve of compound **V** showed the first stage of the mass loss at 30–110°C,

Scheme 1.



X = Cl, 4-EtOOCC$_6$F$_4$.

accompanied by the endothermic effect in the DTA curve (Fig. 1). The mass loss in the first stage was 7.6%, corresponding to the elimination of seven water molecules [the calculated mass fraction of water in $Tb_2(H_2O)_4L_3 \cdot 3H_2O$ was 7.9%]. The dehydrated compound was stable up to 300°C, and then the second stage of the mass loss followed; that process was accompanied by the exothermic effect in the DTA curve and was thus assigned to the compound decomposition. The thermal behavior of compound **VI** was similar; the mass loss in the first stage (30–140°C) accompanied by the endothermic effect was 7.9% [the calculated water fraction in $Eu_2(H_2O)_4L_3 \cdot 3H_2O$ was 8.0%]. The onset of the second stage assigned to the decomposition of the dehydrated compound was observed at 280°C; that stage was accompanied by the exothermic effect.

Elemental analysis data for compounds **VII** and **VIII** corresponded to the $Ln_2(phen)_2(L)_3 \cdot 2H_2O$ formula. The mass loss in the first stage of decomposition of compound **VII** (30–170°C, cf. Fig. 2) was 5.5%, significantly exceeding the content of water in the compound of the suggested formula (1.9%). The second stage of the decomposition was observed at 170–260°C. The total mass loss during the two decomposition stages was 20.5%, close to the calculated sum of water and phenanthroline content in compound **VII** (21.2%). Evidently, the first stage of the compound thermolysis corresponded to dehydration and partial elimination of phenanthroline, the organic ligand elimination ongoing in the second stage of the decomposition. Further decomposition of the complex took place above 260°C. The thermolysis of compound **VIII** occurred similarly: the mass loss in the first decomposition stage (30–180°C) was 5.4%, the second stage was observed at 180–270°C (the total mass loss in the two stages was 19.0%, close to the calculated sum of the fractions of water and phenanthroline, 21.3%), and further decomposition was observed above 270°C. The final stages of decomposition of compounds **VII** and **VIII** were accompanied by the exothermic effects in the DTA curves.

IR spectrum of the H_2L acid contained a broad absorption band at 3200–2300 cm^{-1}, typical of the carboxylic acids associated via the hydrogen bonding. The ν(C=O) band of the acid was found at 1721 cm^{-1} (the similar band in IR spectrum of the acid H_2L' was observed at 1704 cm^{-1} [15]). Table 2 lists the major vibration frequencies of compounds **V**–**VIII**. IR spectra of compounds **V** and **VI** were similar and had much in common with that of the $[Tb_2(H_2O)_4(L')_3 \cdot$

Table 1. Elemental analysis of compounds **V**–**VIII**

Comp. no.	Found, %				Formula	Calculated, %			
	C	H	F	N		C	H	F	N
V	31.8	0.5	28.6		$C_{42}H_{14}F_{24}O_{19}Tb_2$	31.6	0.9	28.6	
VI	30.8	0.8	28.3		$C_{42}H_{14}Eu_2F_{24}O_{19}$	31.9	0.9	28.8	
VII	42.6	0.9	24.0	3.0	$C_{66}H_{20}F_{24}N_4O_{14}Tb_2$	42.2	1.1	24.5	3.0
VIII	41.3	0.9	24.3	3.0	$C_{66}H_{20}Eu_2F_{24}N_4O_{14}$	42.8	1.1	24.6	3.0

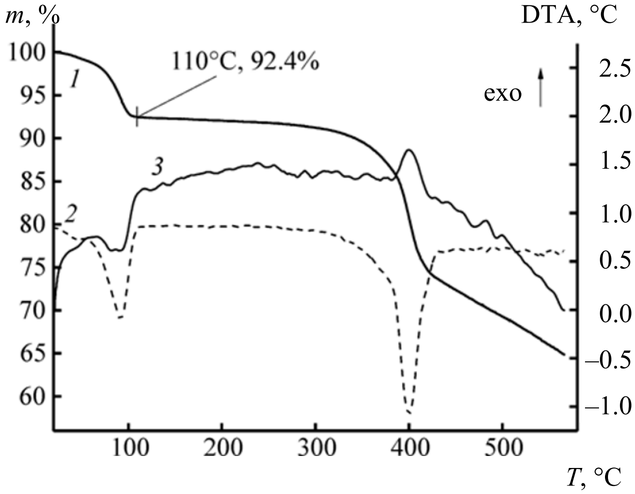


Fig. 1. Thermal analysis of compound **V**: (*1*) TG, (*2*) DTG, and (*3*) DTA.

$2H_2O]_n$ complex characterized by X-ray diffraction analysis [14]. IR spectra of the complexes **VII** and **VIII** were similar as well.

Narrow bands at 3650 and 3644 cm^{-1} and broad strong bands at 3391 and 3363 cm^{-1} in the spectra of complexes **V** and **VI** were assigned to the ν(O–H) vibrations of crystallization and coordinated water molecules, respectively. The spectra of compounds **VII** and **VIII** contained broad bands at 3360 cm^{-1} in the range of the ν(O–H) vibrations assigned to the vibrations of crystallization water molecules associated via the hydrogen bonds.

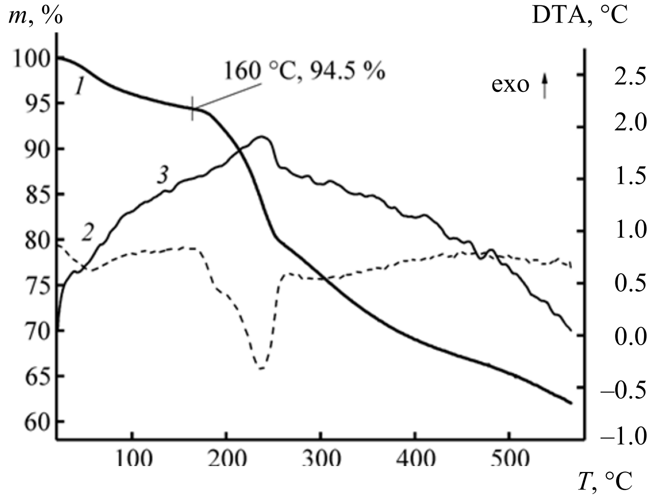


Fig. 2. Thermal analysis of compound **VII**: (*1*) TG, (*2*) DTG, and (*3*) DTA.

IR spectra of compounds **V** and **VI** exhibited strong bands at 1616–1555 and 1472–1394 cm^{-1} due to the ν_{as}(COO) and ν_s(COO) vibrations of coordinated COO^- groups; similar bands were found at 1622–1619 and 1466–1464 cm^{-1} in the spectra of compounds **VII** and **VIII**. The strong band observed at 1504 cm^{-1} in the phenanthroline spectrum was shifted towards higher energy range (1517 cm^{-1}) in the spectra of complexes **VII** and **VIII** due to the coordination of nitrogen atoms at the Ln ions.

The absorption bands at 500–280 cm^{-1} in the spectra of compounds **V–VIII** were assigned to the

Table 2. Main vibration frequencies (cm^{-1}) in IR spectra of complexes **V–VIII**

V	**VI**	**VII**	**VIII**	Assignment
3650	3644			ν(O–H)
3391	3363	3360 br	3360 br	
1616	1613	1622	1619	$\nu_{as}(COO^-)$
1592	1560			
1555				
		1517	1517	phen
1472	1468	1464	1466	$\nu_s(COO^-)$
1406	1394			
490 sh	490 sh			ν(Ln–O)(H_2O)
470	468	471	468	
413	413	414	413	ν(Ln–O)(COO)
332	332	328	327	
292	292	287	287	
		219	215	ν(Ln–N)

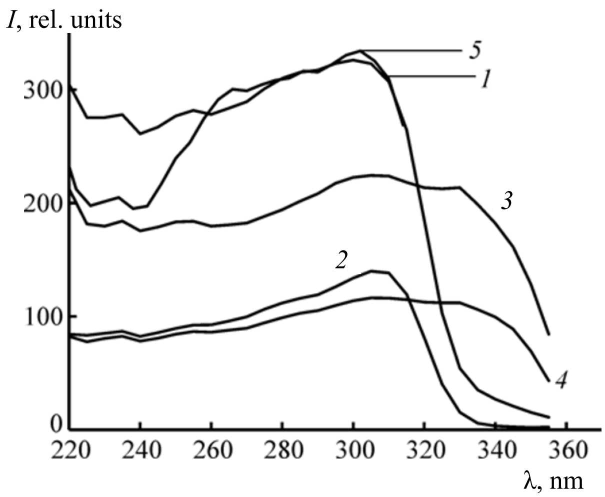


Fig. 3. Excitation spectra of complexes (*1*) **V**, (*2*) **VII**, (*3*) **VI**, (*4*) **VIII,** and (*5*) of the acid H_2L.

Ln–O(COO) and Ln–$O(H_2O)$ (**V** and **VI**) vibrations; the bands at 219 and 215 cm^{-1} were assigned to the ν(Ln–N) vibrations.

The positions of the strongest lines in the X-ray diffraction patterns of compounds **V** and **VI** as well as of compounds **VII** and **VIII** were close, evidencing the identical phase composition of the samples.

V	VI	VII	VIII
6.48	6.48	5.92	5.94
9.82	9.76	8.40	8.46
10.04	10.03		
12.98	12.96	11.78	11.84

To conclude, X-ray diffraction, thermal analysis, and IR spectroscopy results pointed at the pairwise similarity of the compounds **V** and **VI** as well as **VII** and **VIII**. Crystals of the earlier prepared complex of Tb(III) with anions of the H_2L' acid, $Tb_2(H_2O)_4(L')_3\cdot 2H_2O$, were constituted by the layers of coordination 2D polymer $[Tb_2(H_2O)_4(L')_3]_n$ (coordination node TbO_9) containing the bridging anions $(L')^{2-}$ and the hydrate water [14]. Compounds **V** and **VI** were likely coordination polymers as well, of the $Ln_2(H_2O)_4(L)_3\cdot 3H_2O$ composition. Probably, the higher amount of crystallization water molecules in complexes **V** and **VI** as compared to the above-mentioned complex $Tb_2(H_2O)_4(L')_3\cdot 2H_2O$ was due to another packing of the polymeric layers containing longer bridges of the L^{2-} anions. Compounds **VII** and **VIII** $Ln_2(phen)_2(L)_3\cdot 2H_2O$ were likely coordination polymers as well, with the Ln^{3+} ions coordinating two phenanthroline molecules instead of four water ones.

The excitation spectrum of the H_2L acid contained a pair of bands at λ_{max} = 265 and 300 nm (Fig. 3). The excitation at λ_{max} 300 nm induced strong photoluminescence at λ_{max} = 337 nm. The excitation spectra at 250–360 nm were recorded for the complexes as well (Fig. 3); their photoluminescence spectra were registered at the excitation wavelength of 300 nm. The photoluminescence spectra of compounds **V** and **VII** contained narrow bands at λ_{max} = 488, 544, 584, and 620 nm (assigned to the $^5D_4 \rightarrow {}^7F_6$, $^5D_4 \rightarrow {}^7F_5$, $^5D_4 \rightarrow {}^7F_4$, and $^5D_4 \rightarrow {}^7F_3$ transitions in the Tb^{3+} ion) (Fig. 4). The "green" band at λ_{max} 544 nm was the strongest one. The bands positions were almost the same as those in the photoluminescence spectra of the $[Tb_2(H_2O)_4(L')_3\cdot 2H_2O]_n$ and $Tb_2(phen)_2(L')_3\cdot 2H_2O$ complexes [14].

Photoluminescence spectra of compounds **VI** and **VIII** contained the narrow bands at λ_{max} = 590, 614, the broadened band at λ_{max} = 651 nm, and the split band at λ_{max} = 680 and 695 nm, assigned to the $^5D_0\rightarrow{}^7F_1$, $^5D_0\rightarrow{}^7F_2$, $^5D_0\rightarrow{}^7F_3$, and $^5D_0\rightarrow{}^7F_4$ transitions in the Eu^{3+} ion (Fig. 4). The strongest emission band was the "red" one at λ_{max} = 614 nm. The bands positions were close to those in the $Eu_2(L')_3(DEF)_2(EtOH)_2\cdot 2(DEF)$ photoluminescence spectrum (DEF = diethylformamide): λ_{max} = 590, 615, 650, and 695 nm [13].

The emission bands in the spectra of compounds **VII** and **VIII** containing phenanthroline molecules were weaker than those in the spectra of compounds **V** and **VI**. That result contradicted the available data on photoluminescence of the $[Tb_2(H_2O)_4(L')_3\cdot 2H_2O]_n$, $Tb_2(phen)_2(L')_3\cdot 2H_2O$ [14], $Eu_2(H_2O)_4(L')_3\cdot 2H_2O$, and $Eu_2(phen)_2(L')_3\cdot 2H_2O$ [15]: in the latter group, the compounds containing phenanthroline ligand exhibited stronger photoluminescence. The origin of the observed discrepancy requires further investigation.

To conclude, we demonstrated the easy preparation of new photoluminescent complexes of lanthanides with perfluorinated aromatic ligands: anions of octafluorobiphenyl-4,4'-dicarboxylic acid.

EXPERIMENTAL

The following chemicals were used: phenanthroline monohydrate ("analytically pure"), $TbCl_3\cdot 6H_2O$ and $EuCl_3\cdot 6H_2O$ (both "pure"), concentrated aqueous ammonia solution, organic solvents ("analytically pure"), and rectificate EtOH.

NMR spectra were recorded using a Bruker AV-300 instrument [282.4 (^{19}F) and 300 (^{1}H) MHz]. IR spectra were registered using Bruker Tensor 22, Scimitar FTS 2000, and Vertex 80 spectrometers. Electronic spectra were obtained using a Hewlett Packard 8453 instrument. Molecular mass and elemental compositions of the products were determined by means of high-resolution mass spectrometry using a DFS instrument (nominal ionization energy of 70 eV). GC analysis was performed using a Hewlett Packard HP 5980 instrument equipped with an HP-5 quartz capillary column [30 m × 0.52 mm × 2.6 μm, poly(dimethyl/diphenyl siloxane) as the stationary phase] and a heat conductivity detector. Melting points were determined using a Koeffler heating block.

The content of C, H, N, and F in compounds **V**–**VIII** was determined as described in [14]. Thermal analysis was performed in helium atmosphere using a TG 209 F1 Iris ® NETZSCH microthermobalance (specimen mass 10 mg, Al crucible, gas flow 60 mL/min, heating rate 10 deg/min). X-ray diffraction analysis of polycrystals of compounds **V**–**VIII** was carried out using a Shimadzu XRD-7000 diffractometer (CuK_α radiation, Ni filter, 2θ range 5°–60°, 2θ scan step 0.03°, accumulation time 1 s). The polycrystals to be studied were ground in an agate mortar in the presence of heptane, and the so obtained suspension was applied onto the polished side of the standard quartz cell. After heptane was evaporated, a 100 μm even layer of the substance was obtained. Similarly treated polycrystalline silicon was used as external reference.

Spectra of excitation and photoluminescence of solid compounds **V**–**VIII** were registered using a Cary Eclipse Varian fluorescent spectrophotometer at 300 K, the measurement conditions being the same for all samples (V = 500 V, aperture 5 nm).

Ethyl pentafluorobenzoate **I** was prepared as described in [21].

Diethyl octafluorobiphenyl-4,4'-dicarboxylate (III). 13.42 g (99.8 mmol) of $CuCl_2$ was added to a solution of the organozinc reagent **II** prepared from 19.98 g (83.2 mmol) of compound **I**, 16.32 g (249.6 mmol) of Zn powder, 1.58 g (8.32 mmol) of $SnCl_2$, and 45 mL of anhydrous DMF [19]; the mixture was stirred during 6 h at room temperature. Then the reaction mixture was diluted with 300 mL of water, the product was extracted with CH_2Cl_2 (3 × 50 mL), the organic solutions were separated and dried over $MgSO_4$. After the solvent was distilled off, the formed precipitate was filtered off, washed with cold ethanol (50 mL), and dried in air. Yield 14.20 g (77%). 12.66 g (yield 69%) of ester **III** was obtained after recrystallization from ethanol. 1.99 g of ester **III** with purity of 98% (GC) was additionally obtained from the ethanolic mother liquor after the solvent evaporation and sublimation of the product (135–140°C, ≈4 mmHg). Total yield of compound **III** 80%, mp 75.5–76.0°C (EtOH). IR spectrum (KBr), ν, cm^{-1}: 3464, 3009, 2993, 2951, 2912, 1745 (C=O), 1653, 1493, 1473, 1389, 1369, 1304, 1254, 1213, 1018, 978, 860, 717. UV spectrum (EtOH), λ_{max}, nm (log ε): 243 (4.26). ^{1}H NMR spectrum [$(CD_3)_2CO + CCl_4$], δ, ppm: 1.44 t (6H, CH_3, J 7 Hz), 4.47 q (4H, CH_2, J 7 Hz). ^{19}F NMR spectrum [$(CD_3)_2CO + CCl_4$], δ_F, ppm: 23.5 m (4F), 25.2 m (4F). Mass spectrum, m/z: 442.0441 $[M]^+$. Found, %: C 49.02; H 2.30; F 34.33. $C_{18}H_{10}F_8O_4$. Calculated, %: C 48.88; H 2.28; F 34.37. M 442.0446.

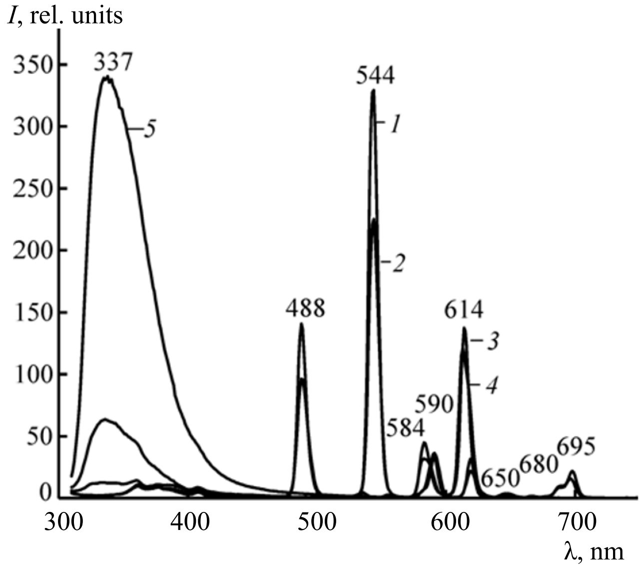


Fig. 4. Photoluminescence spectra of complexes (*1*) **V**, (*2*) **VII**, (*3*) **VI**, (*4*) **VIII,** and (*5*) of the acid H_2L.

Octafluorobiphenyl-4,4'-dicarboxylic acid, H_2L (IV). 8.16 g (145.5 mmol) of KOH was added to a solution of 12.87 g (29.1 mmol) of ester **III** in 195 mL of aqueous EtOH (50 vol %), and the mixture was heated at 90–95°C at stirring during 28 h. The reaction mixture was diluted with water, and the admixtures were extracted with CH_2Cl_2 (3 × 50 mL). The aqueous fraction was acidified with 6 mol/L aqueous HCl. The precipitate was filtered off, washed with water, and dried in a desiccator over P_2O_5. Yield 10.50 g (94%), mp 316–318°C (mp 318–320°C [22]). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 8.51 br. s (2H, COOH). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: 23.7 m (4F), 25.0 m

(4F) [23]. Found, %: C 43.23; H 0.52; F 39.26. $C_{14}H_2F_8O_4$. Calculated, %: C 43.55; H 0.52; F 39.36.

$Tb_2(H_2O)_4L_3·3H_2O$ (V) and $Eu_2(H_2O)_4L_3·3H_2O$ (VI). 2.5 mL of ammonia solution was added to a solution of 0.5 mmol of $LnCl_3·6H_2O$ (Ln = Tb or Eu) (0.185 and 0.183 g, respectively) in 15 mL of water. After 2 h, the $Ln(OH)_3$ precipitate was filtered off on a glass frit filter and washed with water till neutral reaction of the filtrate. The wet precipitate was transferred into a beaker, and 0.29 g (0.75 mmol) of acid **IV** was added. The mixture was stirred till dissolution, and then 30 mL of a 3 : 1 acetone–MeOH mixture was added; white precipitate was formed. The mixture was stirred during 2 h and left overnight. The precipitate was filtered off on a dense paper filter, washed with EtOH and pentane, and dried in air. Yield 0.28 g (70%) (**V**) and 0.36 g (90%) (**VI**).

$Tb_2(phen)_2(L)_3·2H_2O$ (VII) and $Eu_2(phen)_2(L)_3·2H_2O$ (VIII). 0.29 g (0.75 mmol) of acid **IV** and 0.09 g (0.5 mmol) of phen·H_2O were added to the $Ln(OH)_3$ precipitate prepared as described above. The mixture was stirred till dissolution, and then 30 mL of a 3 : 1 acetone–MeOH mixture was added; white precipitate was formed. It was filtered off on a dense paper filter, washed with EtOH and pentane, and dried in air. Yield 0.41 g (88%) (**VII**) and 0.40 g (84%) (**VIII**).

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