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Lanthanide Coordination Polymers Based on Dicyanamide Ligand

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Abstract—The use of dicyanamide and chelating phenanthroline ligands resulted in the synthesis of organometallic europium and neodymium coordination polymers $[Ln(Phen)_2\{N(CN)_2\}_{3-x}Cl_x(CH_3OH)]$-$(CH_3OH)_y]_\infty$ (Ln = Eu (**I**), Nd (**II**)) in which the dicyanamide ligand forms 1D chains, while the phenanthroline ligand provides packing of the 1D polymer in the crystal lattice. Under similar conditions, the binuclear complex $[Gd(Phen)_2(\mu\text{-}OH)\{N(CN)_2\}(H_2O)]_2Cl_2$ (**III**) was obtained for gadolinium, with the dicyanamide ligands being coordinated to only one gadolinium ion without forming a polymer chain (CCDC CIF files nos. 1936378 (**I**), 1936379 (**II**), 1936377 (**III**)).

Keywords: rare earth elements, coordination polymer, X-ray diffraction, luminescence

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INTRODUCTION

Organometallic coordination polymers (OMCPs) of lanthanides are of considerable interest both for fundamental research as examples of coordination compounds of rare earth elements with unusual environment and for applied purposes as they can serve for the development of promising luminescent materials [1, 2].

The combination of luminescent properties of lanthanide ions, in combination with the unique structural features of OMCPs opens up new opportunities for the design of luminescent materials. The use of isostructural OMCPs provides for doping with various lanthanides in specified ratios for fine tuning of luminescent and magnetic properties [2–4].

The purpose of this study is to obtain luminescent organometallic coordination polymers able to emit in both visible and IR ranges. The fabrication of these polymers is usually a challenging task, because of easy luminescence quenching by the groups present in the organic ligand and capable of high-frequency vibrations (O–H, N–H, C–H) [1].

An especially important issue in the design of coordination compounds of this type is the choice of ligand that acts as a linker assembling the coordination polymer. The dicyanamide ligand, $[N(CN)_2]^-$, seems to be the optimal linker for the design of these systems in view of its electronic and steric features and the absence of hydrogen atoms. Despite the obvious choice of dicyanamide ligand, which forms bridges between the metal cations, the application of this ligand in the lanthanide coordination chemistry for the construction of OMCPs is limited to few examples: $[Nd\{N(CN)_2\}_3(HMPTA)_x]_n$ (x = 2, 3 [5]), $[Ln\{N(CN)_2\}_3(H_2O)(L)_2]$ (Ln = Gd, L = bipyridine [6], phenanthroline [7]; Ln = Sm, L = bipyridine [6]), $[Ln\{N(CN)_2\}_2(H_2O)_3(L)_2]^+[N(CN)_2]^-(L)$ (Ln = La, Pr, Sm; L = phenanthroline) [6], and $[Ln\{N(CN)_2\}_3(H_2O)_2]$ (Ln = Eu, Tb [8]).

Here we report the preparation of heteroligand europium and neodymium complexes with dicyanamide and phenanthroline ligands, which form 1D coordination polymers, and their crystal structure and photophysical properties.

EXPERIMENTAL

Reagent grade lanthanide chloride hexahydrates $LnCl_3(H_2O)_6$ and sodium dicyanamide (96%, Sigma-Aldrich) were used as received. High-purity grade methanol was dehydrated by refluxing over magnesium metal; analytical grade 9,10-phenanthroline (Phen) (Chemapol) was purified by vacuum sublimation.

Synthesis of $[Eu(Phen)_2\{N(CN)_2\}_{3-x}Cl_x(CH_3OH)_x](CH_3OH)$ (I). A solution of sodium dicyanamide (0.134 g, 1.5 mmol) in dry methanol (4 mL) was added with stirring to a solution of $EuCl_3 \cdot 6H_2O$ (0.183 g, 0.5 mmol) in dry methanol (4 mL), and the mixture was stirred for 1 h. A solution of Phen in dry methanol (0.180 g, 1.0 mmol) was added with vigorous stirring to the resulting reaction mixture and the mixture was heated to boiling and then stirred at room temperature for 5 h. The solution was separated from the precipitate and slowly concentrated to ~1 mL. The resulting crystals were separated from the solution and dried in vacuo. The yield of **I** was 0.253 g (68%).

For $C_{30.81}H_{20.76}N_{12.43}O_{1.19}Cl_{0.19}Eu$

Anal. calcd., %	C, 49.79	H, 2.80	N, 23.39
Found, %	C, 48.69	H, 1.68	N, 23.78

Synthesis of $[Nd(Phen)_2\{N(CN)_2\}_{3-x}Cl_x(CH_3OH)_x](CH_3OH)$ (II) was carried out by a similar procedure from $NdCl_3 \cdot 6H_2O$ (0.359 g, 1.0 mmol), sodium dicyanamide (0.268 g, 3.0 mmol), and Phen (0.360 g, 2.0 mmol). The yield of **II** was 0.353 g (48%).

For $C_{30.45}H_{22.2}N_{11.35}O_{1.55}Cl_{0.55}Nd$

Anal. calcd., %	C, 49.66	H, 3.04
Found, %	C, 49.10	H, 2.89

An attempt to prepare the corresponding gadolinium complex under similar conditions resulted in a mixture of compounds, with the binuclear complex $[Gd(Phen)_2(\mu\text{-}OH)\{N(CN)_2\}(H_2O)]_2Cl_2$ (**III**) being the only isolated product, evidently formed upon hydrolysis with coordinated water present in the starting gadolinium chloride hexahydrate. Only several crystals of **III** were isolated from the reaction mixture.

Single crystal X-ray diffraction analysis of **I**, **II**, and **III** was carried out on a SMART APEX2 CCD diffractometer (MoK_α radiation, graphite monochromator, ω-scan mode). The structures were solved by the direct methods and refined by least-squares method in the full-matrix anisotropic approximation on F^2_{hkl}. In the refinements of structures **I** and **II**, positional and thermal constraints were used for atoms corresponding to superposition of the $N(CN)_2$ and $Cl\cdots HOCH_3$ moieties. At the final stage of refinement, the constraints on the Ln–Cl and Ln–N bond lengths (Ln = Eu, Nd) were eliminated and the occupancies for the disordered moiety of **I** were fixed. The Cl^- occupancies in **I** and **II** are 0.19 and 0.55, respectively. Analysis of the difference Fourier maps for the crystal of **III** demonstrated that the central nitrogen atom of $N(CN)_2$ is disordered over two positions with occupancies of 0.52 and 0.48. The hydrogen atoms of Phen and the disordered CH_3OH molecule were calculated geometrically, while the hydrogen atom of the coordinated methanol molecule was located from difference Fourier maps. All calculations were performed using the SHELXTL PLUS software package [9]. The main crystallographic data and structure refinement parameters for compounds **I**–**III** are summarized in Table 1.

The atomic coordinates and other structure parameters are deposited with the Cambridge Crystallographic Data Centre (nos. 1936378 (**I**), 1936379 (**II**); 1936377 (**III**) deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

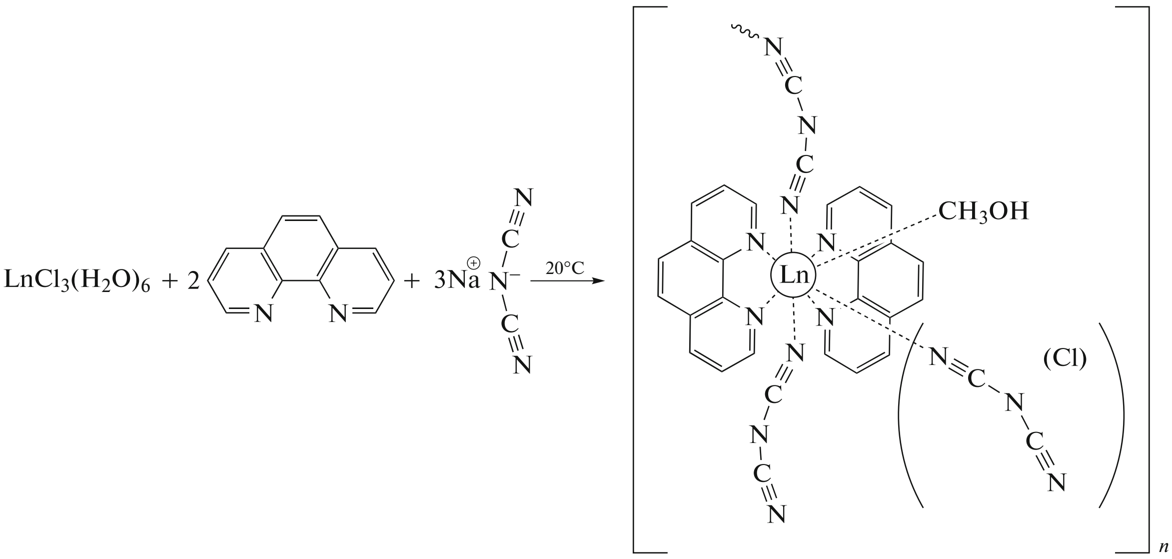
The luminescence spectra were measured on a Horiba-Jobin-Yvon Fluorolog FL 3-22 spectrometer equipped with a 450 W continuous xenon lamp as an excitation source and an R928 photomultiplier tube. The excited state lifetimes of lanthanide ions were measured at their emission maxima and averaged over at least three measurements. The data were processed using the Origin 8.6 software package.

RESULTS AND DISCUSSION

The reactions of solutions of europium and neodymium chlorides $LnCl_3 \cdot 6H_2O$ (Ln = Eu, Nd) in dry methanol with 3 equiv. of sodium dicyanamide and 2 equiv. of Phen gave rise to heteroligand complexes of a variable composition described as $[Ln(Phen)_2\{N(CN)_2\}_{3-x}Cl_x(CH_3OH)]_n$ (Ln = Eu (**I**), Nd (**II**)) in 68 and 48% yields, respectively. Recrystallization from hot anhydrous methanol afforded single crystalline samples of **I** and **II**.

Table 1. Crystallographic data and structure refinement details for compounds **I–III**

Parameter	Value		
	I	**II**	**III**
Molecular formula	$C_{30.81}H_{20.76}N_{12.43}O_{1.19}Cl_{0.19}Eu$	$C_{30.45}H_{22.20}N_{11.35}O_{1.55}Cl_{0.55}Nd$	$C_{56}H_{54}N_{14}O_{8}Cl_{2}Gd_{2}$
M	742.83	735.56	1436.53
T, K	120	120	120
System	Triclinic	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$	$P\bar{1}$
Z	2	2	1
a, Å	8.640(3)	8.6645(6)	10.9608(14)
b, Å	10.017(4)	9.9103(7)	11.0365(14)
c, Å	18.605(8)	18.8326(13)	12.1296(15)
α, deg	96.331(11)	96.4521(15)	95.947(2)
β, deg	93.479(10)	93.5244(16)	103.215(2)
γ, deg	112.824(9)	112.1397(14)	100.012(2)
V, Å^3	1465.7(10)	1478.89(18)	1390.9(3)
ρ(calcd.), g cm^{-3}	1.683	1.652	1.715
μ, cm^{-1}	22.08	18.54	25.28
F(000)	737	732	714
$2\theta_{max}$, deg	58	58	55
Number of measured reflections	9135	17940	6257
Number of unique reflections	7616	7856	6257
Number of reflections with $I > 2\sigma(I)$	6692	6610	5792
Number of refined parameters	422	427	376
R-factors ($I > 2\sigma(I)$)	$R_1 = 0.0427$, $wR_2 = 0.0957$	$R_1 = 0.0384$, $wR_2 = 0.0790$	$R_1 = 0.0317$, $wR_2 = 0.0752$
R-factors (for all reflections)	$R_1 = 0.0499$, $wR_2 = 0.1006$	$R_1 = 0.0513$, $wR_2 = 0.0847$	$R_1 = 0.0371$, $wR_2 = 0.0786$
GOOF	1.029	0.982	1.084
Residual electron density (max/min), e Å^{-3}	1.429/–1.320	1.309/–1.728	1.817/–0.874



The structures of the obtained complexes were established by X-ray diffraction; they can be considered as products of co-crystallization of a lanthanide tris-dicyanamide complex with a bis-dicyanamide chloride complex in ~4 : 1 ratio for **I** and ~1 : 1 ratio for **II**.

In the isostructural complexes **I** and **II**, one dicyanamide ligand occupies a bridging position, combining the molecules into 1D coordination polymer extended along the crystallographic *a* axis (Fig. 1). Due to the presence of bridging $N(CN)_2^-$ anion, the lanthanide coordination number in the crystal is 9 and its coordination polyhedron can be described as a single-capped square antiprism. The roles of the two terminal $N(CN)_2^-$ anions in the supramolecular organi-

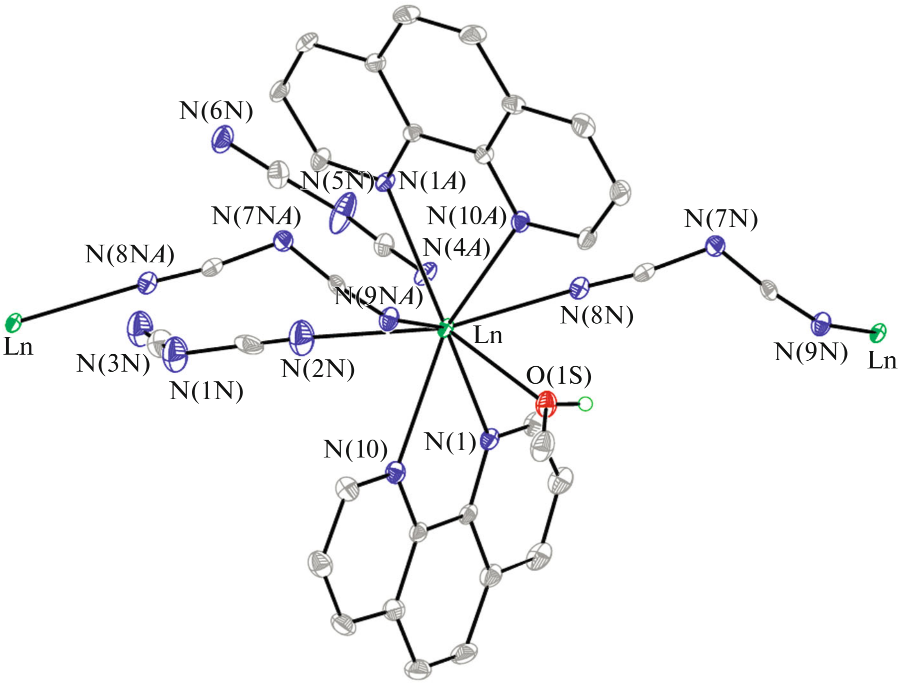


Fig. 1. General view of complexes **I** and **II** illustrating a 1D coordination polymer unit with atoms represented as thermal ellipsoids ($p = 50\%$). The Cl^- ion and CH_3OH solvate molecule are not shown. Selected bond lengths: Eu(1)–O(1S), 2.422(3); Eu(1)–N(9N*A*), 2.483(4); Eu(1)–N(8N), 2.491(4); Eu(1)–N(4N), 2.495(4); Eu(1)–N(2N), 2.523(12); Eu(1)–N(1), 2.591(4); Eu(1)–N(10), 2.641(4); Eu(1)–N(10*A*), 2.701(4); Eu(1)–N(1*A*), 2.702(3); Eu(1)–Cl(1), 2.703(9) Å in **I** and Nd(1)–O(1S), 2.480(3); Nd(1)–N(2N), 2.533(15); Nd(1)–N(4N), 2.533(3); Nd(1)–N(8N), 2.541(3); Nd(1)–N(9N*A*), 2.544(3); Nd(1)–N(1), 2.642(3); Nd(1)–N(10), 2.669(3); Nd(1)–N(1*A*), 2.731(3); Nd(1)–N(10*A*), 2.739(3); Nd(1)–Cl(1), 2.833(2) Å in **II**.

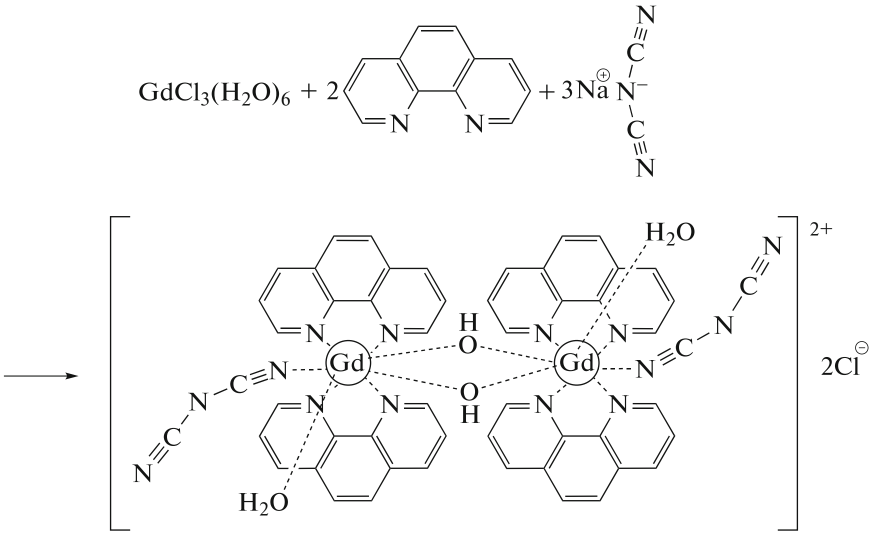
zation of 1D coordination polymers are markedly different. One of them (N(8N)–N(9N)) is involved in fairly strong $CH_3O(1S)–H\cdots N(9N)C$ hydrogen bonds ($O(1S)\cdots N(9N)$ 2.793(2) Å), resulting in the formation of layers with a coating formed by phenanthroline ligands. The second $N(CN)_2^-$ ligand (N(2N)–N(3N)) actually does not form strong contacts, except for $C–H\cdots N$. Apparently, this type of interactions is responsible for superposition of Cl^- and $N(CN)_2^-$ anions for this ligand (Fig. 2). The replacement of $N(CN)_2^-$ by chloride anion does not give rise to cavities in the crystals of **I** and **II**, because Cl^- is located together with an additional methanol solvate molecule involved in the $Cl\cdots OH$ contact (Fig. 2). It is noteworthy that the $N(CN)_2^- : Cl^-$ ratio in **I** and **II** is not a function of the lanthanide ion radius. While attempting the synthesis of a coordination polymer devoid of the chloride ion, we studied a number of crystals, each containing the above-indicated superposition, but the $N(CN)_2^- : Cl^-$ ratios were different and the choice of particular samples was dictated by only the quality of diffraction data sets. This suggests that the considered type of complexes is characterized by a broad range of $N(CN)_2^- :$ Cl^- ratios for which the indicated type of packing is retained.

Apart from the H-bonds, which combine 1D chains into layers, stacking interactions between the phenanthroline ligands with a distance of ~3.4 Å between the ring planes are also present in the crystals of **I** and **II**. It is of interest that the stacking interactions occur only for one ligand, while for the second ligand, the corresponding distances exceed the sum of the van der Waals radii. Furthermore, particularly the phenanthroline ligand (N(1)–N(10)) that participates in the stacking interactions forms stronger bonds (2.642(3)–2.669(3) Å versus 2.731(3)–2.739(3) Å) in **II**.

In view of the fact that the indicated lanthanide ion distances influence the efficiency of luminescence sensitization, it can be concluded that stacking interactions can not only give rise to additional states [10, 11], but also increase the binding efficiency of the lanthanide ion with the ligand.

No complete replacement of the chloride ion in the coordination sphere was achieved even when the reaction was carried out for a longer time and with a 5% excess of sodium dicyanamide. Apparently, the nucleophilicity of the dicyanamide ligand is too low for exhaustive displacement of the chloride ligands. In the case of gadolinium, the binuclear complex $[Gd(Phen)_2(\mu\text{-}OH)\{N(CN)_2\}(H_2O)]_2Cl_2$ (**III**) proved to be the only isolated product.

The structure of **III** was also confirmed by X-ray diffraction. Unlike complexes **I** and **II**, complex **III** is a dimer bridged with hydroxyl groups, the dicyanamide ligands are coordinated to only one gadolinium ion and do not form a polymer chain (Fig. 3). Compound **III** crystallizes as a solvate with four methanol molecules. Complex **III** in the crystal is located at the center of symmetry. The coordination polyhedron of gadolinium is a distorted square antiprism. The outer-sphere chloride anion is hydrogen-bonded to the bridging OH group ($O\cdots Cl$ 3.251(3) Å) and solvate methanol and coordinated water molecules ($O\cdots Cl$ 3.119(3)–3.213(5) Å). This solvent-separated ion pair is combined into H-bonded chains via the $O–H\cdots N$ hydrogen bonds formed by water molecules and dicyanamide ligand ($O\cdots N$ 2.782(5) Å). An interesting feature of complex **III** is a shortened intramolecular contact between phenanthroline ligands (the distance between the ligand planes is 3.401 Å; the shortest $C\cdots C$ contact is 3.542(4) Å), which suggests the occurrence of intramolecular stacking interactions in **III**.



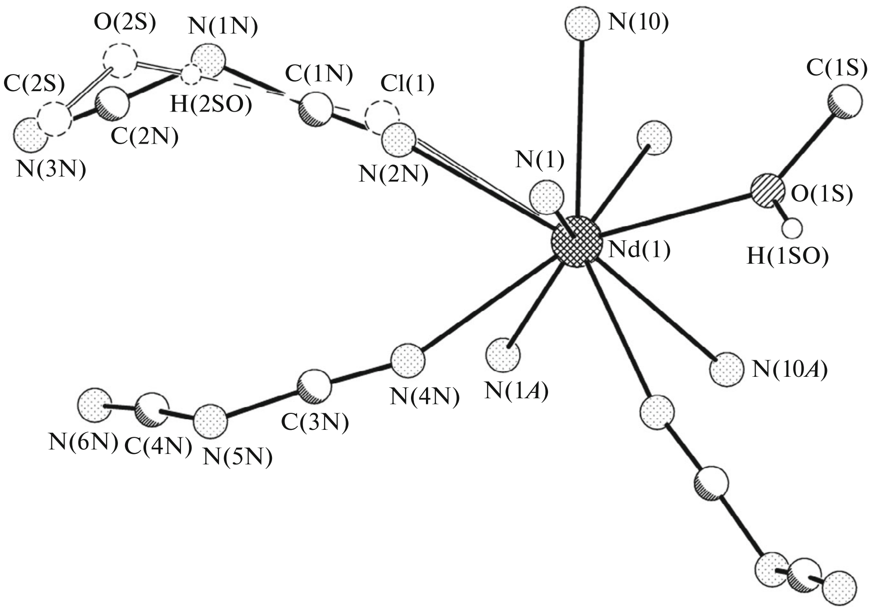


Fig. 2. Scheme illustrating the superposition of $Cl^{-}\cdots HOCH_3$ and $N(CN)_2$ moieties in the crystal of **II**. The carbon atoms of phenanthroline ligands are omitted.

It is known that, because of specific electronic structure of 4*f* elements, direct excitation of Ln^{3+} ions usually does not result in intense luminescence [12]. Therefore, luminescence of lanthanide ions is usually sensitized by means of organic ligands coordinated to Ln^{3+} ions and able to transfer the absorbed electromagnetic energy from the ligand to the Ln^{3+} ion (antenna effect) [13]. In the obtained complexes **I** and **II**, both the dicyanamide and phenanthroline ligands could serve as such antennas. Luminescence is known for both phenanthroline [14] and homoligand dicyanamide [8] lanthanide complexes.

The luminescence spectrum of europium complex **I** shows narrow lines corresponding to Eu^{3+} characteristic transitions, $^5D_0 \rightarrow {}^7F_J$ ($J = 0$–4) and $^5D_1 \rightarrow {}^7F_J$ ($J = 0$–2) (Fig. 4). The $^5D_0$–$^7F_0$ electron transition forbidden by the selection rules (575–585 nm range) gives rise to one weak symmetrical line, indicating the presence of one sort of europium environment. The luminescence decay kinetics is described by a monoexpotential law, which also attests to the presence of only one luminescence center. The lifetimes of the $^5D_0$ state were 0.60 ± 0.02 ms, which is in good agreement with the established composition of the coordination sphere. In the range of $^5D_0$–$^7F_1$ magnetic dipole transition (590–600 nm) of the luminescence spectrum of complex **I**, only one, relatively broad band is present. In a centrosymmetric environment, the $^5D_0$–$^7F_1$ magnetic dipole transition predominates, and the $^5D_0$–$^7F_2$: $^5D_0$–$^7F_1$ integrated intensity ratio is <1, whereas symmetry distortion of the environment of the europium ion increases the intensity of the $^5D_0$–$^7F_2$ transition. The calculated ratio for the complex is ~4.5, which reveals a symmetry of the charge environment of the europium ion substantially remote from the inversion center. In particular, in the case of europium β-diketonates, this value is 14–20. The number of Stark components of other $^5D_0$–^7F_J transitions ($J = 2$–4) is also close to the maximum possible value, which indicates a pronounced distortion of the ion symmetry. Meanwhile, moderate overall splittings of the transitions attest to an axial arrangement of charges, which is in line with X-ray diffraction data.

The luminescence spectra of neodymium complex **II** were also recorded and demonstrated luminescence in the near-IR region with the $^4F_{3/2}$–$^4I_{11/2}$ transition (1050–1100 nm) being the most intense (Fig. 5).

According to the luminescence sensitization concept, the energy absorbed by the ligand is transferred through the singlet ($^1\pi\pi^*$) and triplet ($^3\pi\pi^*$) states to the excited resonance levels of the lanthanide ion. The luminescence excitation spectra of complexes **I** and **II** exhibit broad bands for the ligands in the 250–400 nm range, apart from narrow bands for *f*–*f* transitions of Ln^{3+} (Fig. 6). Relying on the spectral data for the previously studied lanthanide complexes of 1,10-phenanthroline, the band with a maximum at 350 nm (28 570 cm^{-1}) can be assigned to the S_1 state of the phenanthroline ligand (Fig. 6). A comparison of the luminescence excitation spectra of isostructural Eu and Nd complexes shows an additional broad band with a maximum at 400 nm (25 000 cm^{-1}), which is missing for neodymium. The long-wavelength edge of

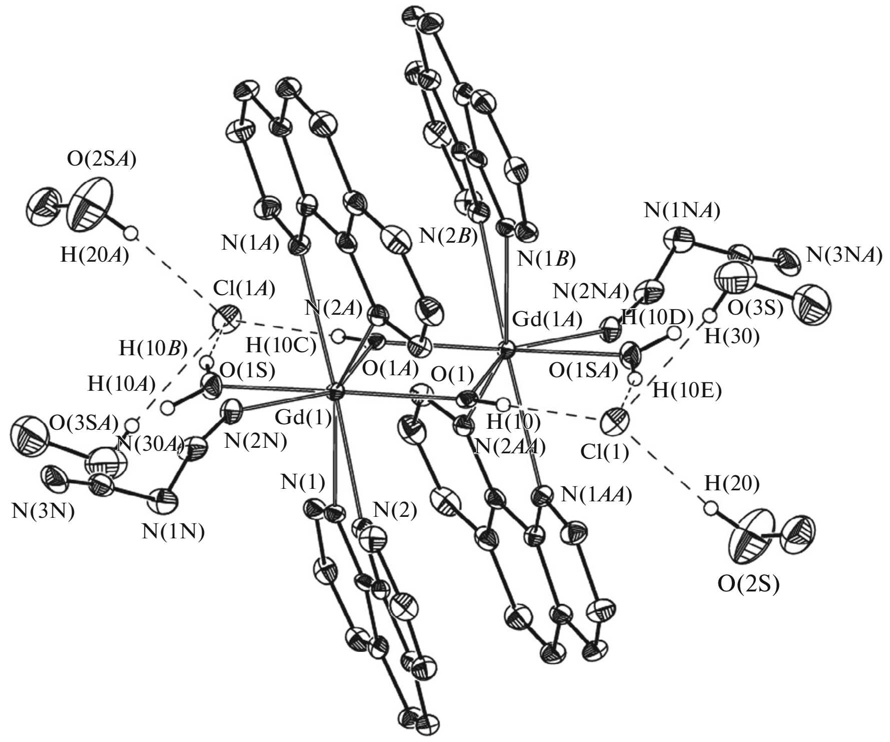


Fig. 3. General view of complex **III** with atoms represented as thermal ellipsoids ($p = 50\%$). Selected bond lengths: Gd(1)–O(1), 2.258(3); Gd(1)–O(1*A*), 2.300(3); Gd(1)–O(1S), 2.411(3); Gd(1)–N(2N), 2.501(3); Gd(1)–N(1*A*), 2.579(3); Gd(1)–N(1), 2.601(3); Gd(1)–N(2*A*), 2.640(4); Gd(1)–N(2), 2.640(4); Gd(1)···Gd(1), 3.7046(5) Å.

this band extends almost to 500 nm. This comparison allows this band to be interpreted as a ligand-to-metal charge transfer (LMCT) band.

The position of the charge transfer band can be estimated using the following expression valid for a large number of compounds:

$$E_{\mathrm{CTS}} \approx 30000[\chi_{\mathrm{opt}}(\mathrm{X}) - \chi_{\mathrm{uncorr}}(\mathrm{M})]\ \mathrm{cm}^{-1}, \qquad (1)$$

where $\chi_{\mathrm{opt}}(\mathrm{X})$ is the optical electronegativity of the ligand, $\chi_{\mathrm{uncorr}}(\mathrm{M})$ is the lanthanide ion electronegativity without allowance for spin correlation. The optical electronegativity varies in the series of halides similarly to the Pauling electronegativity. Since the energy of

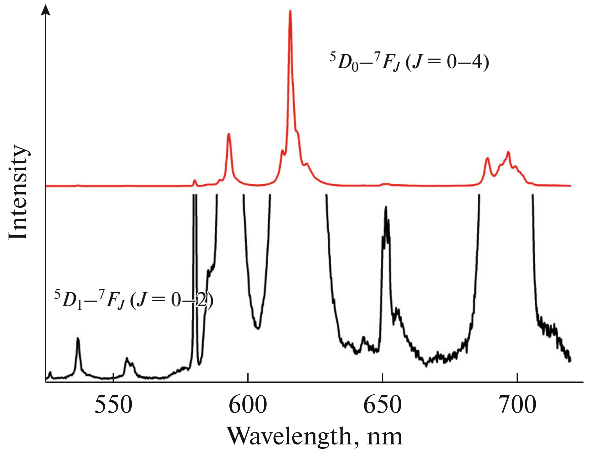


Fig. 4. Luminescence spectra of complex **I**. The black spectrum was recorded with increased intensity. $\lambda_{\mathrm{excit}} = 340$ nm.

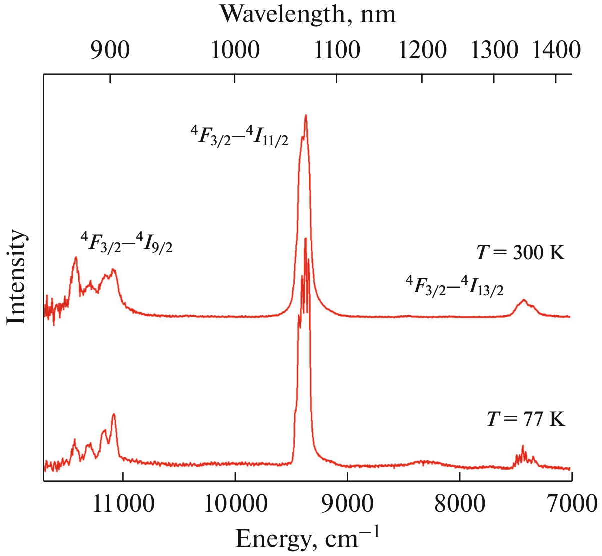


Fig. 5. Luminescence spectra of complex **II** at 300 and 77 K. $\lambda_{\mathrm{excit}} = 340$ nm.

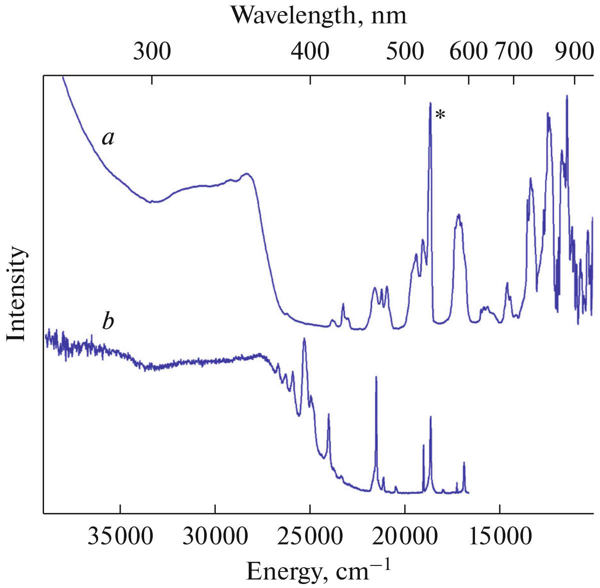


Fig. 6. Luminescence excitation spectra of complexes (*a*) **II** and (*b*) **I** at 300 K.

the LMCT state was determined experimentally, the use of χ_{uncorr}(Eu) = 1.9925 gives χ_{opt}(X) of 2.83. Previously we employed this procedure for determining the optical electronegativity of the ligand in europium chloride complexes with phenanthroline [15]. It was shown that the LMCT state is due to chloride to Eu^{3+} charge transfer. For the dicyanamide complexes considered here, presumably, the greatest contribution to the LMCT state can also belong to the $N(CN)_2$ groups. Indeed, the dicyanamide ligand in complexes **I** and **II** functions as an anionic ligand similar to the halide ion in the related phenanthroline complexes of lanthanides [15]. Thus, being a pseudohalide, dicyanamide is expected to have an optical electronegativity comparable with the optical electronegativity of chlorine (3.1), which we actually found using the estimated χ_{opt}(X) value for **I**.

Thus, as a result of our study, we prepared organometallic coordination polymers with dicyanamide linkers and phenanthroline ligands, efficient sensitizers of lanthanide ion luminescence in the visible and near-IR ranges.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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