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Structural Diversity of Heteroleptic 1,2,4-Triphenylcyclopentadienyl-Bipyridine Complexes of Rare Earth Metals

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Abstract—The reaction of triphenylcyclopentadienyl potassium and bipyridine with lanthanum, praseodymium, erbium, lutetium, and scandium chloride tetrahydrofuranates results in the formation of binuclear $[Cp^{Ph3}Ln(Bipy)Cl(\mu_2\text{-}Cl)]_2$ (Ln = La (**I**), Pr (**II**)) and mononuclear $[Cp^{Ph3}Ln(Bipy)Cl_2(THF)]$ (Ln = Er (**III**), Lu (**IV**)), $[Cp^{Ph3}Sc(Bipy)Cl_2]$ (**V**) complexes (Cp^{Ph3} = 1,2,4-triphenylcyclopentadienyl, Bipy = bipyridine). The decrease in the REE radius in the series La...Sc results in the formation of mononuclear instead of binuclear complexes and in a decrease in the coordination number of the central ion. The coplanar arrangement of two different π-systems gives rise to stacking interactions between the triphenylcyclopentadienyl ligand and bipyridine. The molecular structure of complexes **I**–**V** was established by X-ray diffraction analysis (CCDC nos. 2308609 (**I**), 2308608 (**II**), 2308610 (**III**), 2308611 (**IV**), 2308607 (**V**)).

Keywords: lanthanides, triphenylcyclopentadienyl ligand, bipyridine, X-ray diffraction analysis

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INTRODUCTION

Aryl-substituted cyclopentadienyl ligands play an important role in the design of new types of organometallic compounds of 4f elements [1–6].

Previously, we showed that the combination of a polyphenyl-substituted cyclopentadienyl ligand and a polydentate N-heterocyclic ligand in the same coordination sphere of a rare earth element (REE) makes it possible to obtain mononuclear monocyclopentadienyl complexes based on tri- and tetraphenyl-substituted cyclopentadienyl ligands, which cannot be achieved otherwise [7–9]. We reported the synthesis and structural study of the triphenylcyclopentadienyl and tetraphenylcyclopentadienyl complexes of elements of the middle of 4*f*-series, particularly, Nd, Tb, and Gd complexes with a bipyridine ligand [8]. The structure of type $[(C_5H_{5-n}Ph_n)LnCl_2(Bipy)(THF)_n]$ complexes, which is relatively simpler than that of polynuclear structures, usually formed by monocyclopentadienyl compounds with polyaryl-substituted cyclopentadienyl ligands, enables more detailed analysis of the molecular structure and packing in the crystal lattice for this type of complexes.

The purpose of the present study is to synthesize REE cyclopentadienyl bipyridine complexes: $[Cp^{Ph3}Ln(Bipy)Cl(\mu_2\text{-}Cl)]_2$ (Ln = La (**I**), Pr (**II**)), $[Cp^{Ph3}Ln(Bipy)Cl_2(THF)]$ (Ln = Er (**III**), Lu (**IV**)), and $[Cp^{Ph3}Sc(Bipy)Cl_2]$ (**V**) (Cp^{Ph3} = 1,2,4-triphenylcyclopentadienyl, Bipy = bipyridine) and to identify the differences between their structures depending on the ionic radius of the complex-forming metal.

EXPERIMENTAL

Compounds **I**–**V** were synthesized in the atmosphere of purified argon in anhydrous solvents using a SPECS-GB2 glove box. Tetrahydrofuran was predried over NaOH and distilled from potassium/benzophenone. Hexane was distilled from sodium–potassium eutectic/benzophenone. Toluene was distilled from sodium/benzophenone. $LnCl_3(THF)_n$ (Ln = La, Pr, Er, Lu, Sc) were synthesized by a reported procedure [10]. Benzyl potassium was prepared by a modified published procedure [11]. 1,2,4-Triphenylcyclopentadiene was obtained by a known procedure [12], recrystallized from absolute ethanol, and dried under dynamic vacuum; and bipyridine was sublimed in

vacuo prior to use. The elemental analysis of complexes **I** and **III** was performed on a Thermo Scientific FLASH 2000 CHNS/O analyzer with addition of Co_3O_4 to ensure complete combustion. The elemental analysis of complexes **II**, **IV**, and **V** was carried out on a Leco TruSpec Micro CHNS analyzer.

Synthesis of $[Cp^{Ph3}LaCl_2(Bipy)]_2$ (I). A solution of benzyl potassium (0.034 g, 0.26 mmol) in THF (5 mL) was slowly added with stirring to a solution of $Cp^{Ph3}H$ (0.074 g, 0.25 mmol) in THF (10 mL). The reaction mixture was stirred for 15 min, and the resulting solution of 1,2,4-triphenylcyclopentadienyl potassium was slowly added to a stirred suspension of $LaCl_3(THF)_2$ (0.097 g, 0.25 mmol) in THF (15 mL). The reaction mixture was stirred for 12 h, then a solution of Bipy (0.039 g, 0.25 mmol) in THF (2 mL) was added. The reaction mixture was stirred for 12 h, and the precipitate that formed was separated by centrifugation (4000 rpm, 10 min). The precipitate was extracted with THF (2 × 5 mL) to dissolve the remainder of the complex and centrifuged again. Hexane (40 mL) was carefully added to the combined solutions, so as to avoid mixing of the layers. After 3 days, the crystals of complex **I** formed. The crystals were dried under dynamic vacuum. The yield of compound **I**, as an orange crystalline powder, was 0.098 g (0.074 mmol, 59%).

For $C_{66}H_{50}N_4Cl_4La_2$

Anal. calcd., %	C, 60.11	H, 3.83	N, 4.25
Found, %	C, 59.66	H, 4.23	N, 4.16

The crystals suitable for X-ray diffraction were obtained by slow diffusion of hexane into a solution of **I** in THF. According to X-ray diffraction data, the unit cell of complex **I** contained one THF molecule. This molecule was lost during vacuum drying.

The synthesis of $[Cp^{Ph3}PrCl_2(Bipy)]_2$ (II) was performed by the procedure described above starting from $PhCH_2K$ (0.068 g, 0.52 mmol), $Cp^{Ph3}H$ (0.147 g, 0.5 mmol), $PrCl_3(THF)_2$ (0.196 g, 0.5 mmol), and Bipy (0.078 g, 0.5 mmol). The combined solution of the complex in THF obtained after centrifugation of the reaction mixture was concentrated to 10 mL. Hexane (20 mL) was carefully added to the concentrated solution. The yellow-green powder precipitated after 2 days was dissolved in THF (50 mL), and hexane (50 mL) was carefully added to the solution. After 4 days, the crystals of complex **II** formed. The crystals were dried under dynamic vacuum. The yield of compound **II**, as a yellow-green crystalline powder, was 0.172 g (0.130 mmol, 52%).

For $C_{66}H_{50}N_4Cl_4Pr_2$

Anal. calcd., %	C, 59.93	H, 3.81	N, 4.24
Found, %	C, 59.53	H, 4.10	N, 4.25

The crystals suitable for X-ray diffraction were obtained by slow diffusion of hexane into a solution of **II** in THF. According to X-ray diffraction data, the unit cell of complex **II** contained one THF molecule. This molecule was lost during vacuum drying.

The synthesis of $[Cp^{Ph3}ErCl_2(Bipy)(THF)]$ (III) was performed by the procedure described above for **I** starting from $PhCH_2K$ (0.034 g, 0.26 mmol), $Cp^{Ph3}H$ (0.074 g, 0.25 mmol), $ErCl_3(THF)_3$ (0.122 g, 0.25 mmol), and Bipy (0.039 g, 0.25 mmol). The yield of compound **III**, formed as an orange crystalline powder, was 0.114 g (0.150 mmol, 60%).

For $C_{37}H_{33}N_2OCl_2Er$

Anal. calcd., %	C, 58.49	H, 4.38	N, 3.69
Found, %	C, 58.60	H, 4.39	N, 3.76

The crystals suitable for X-ray diffraction were obtained by slow diffusion of hexane into a solution of **III** in THF.

The synthesis of $[Cp^{Ph3}LuCl_2(Bipy)(THF)]$ (IV) was performed by the procedure described above for **I** starting from $PhCH_2K$ (0.034 g, 0.26 mmol), $Cp^{Ph3}H$ (0.074 g, 0.25 mmol), $LuCl_3(THF)_3$ (0.124 g, 0.25 mmol), and Bipy (0.039 g, 0.25 mmol); the precipitate obtained by centrifugation of the reaction mixture was extracted with THF (4 × 15 mL). The yield of compound **IV**, formed as an orange crystalline powder, was 0.078 g (0.102 mmol, 41%).

For $C_{37}H_{33}N_2OCl_2Lu$

Anal. calcd., %	C, 57.90	H, 4.34	N, 3.65
Found, %	C, 57.67	H, 4.27	N, 3.53

The crystals suitable for X-ray diffraction were obtained by slow diffusion of hexane into a solution of **IV** in THF.

The synthesis of $[Cp^{Ph3}ScCl_2(Bipy)]$ (V) was performed by the procedure described above for **I** starting from $PhCH_2K$ (0.034 g, 0.26 mmol), $Cp^{Ph3}H$ (0.074 g, 0.25 mmol), $ScCl_3(THF)_3$ (0.092 g, 0.25 mmol), and Bipy (0.039 g, 0.25 mmol). The combined solution of the complex in THF obtained after centrifugation of the reaction mixture was concentrated to dryness. The resulting dark powder was washed with toluene (10 mL) and dissolved again in THF (7 mL). Hexane

(20 mL) was carefully added to the solution. After several days, the crystals of complex **V** formed. The crystals were dried under dynamic vacuum. The yield of compound **V**, formed as a yellow crystalline powder, was 0.117 g (0.207 mmol, 83%).

For $C_{33}H_{25}N_2Cl_2Sc$			
Anal. calcd., %	C, 70.11	H, 4.46	N, 4.96
Found, %	C, 69.77	H, 5.09	N, 4.44

The crystals suitable for X-ray diffraction were obtained by slow diffusion of hexane into a solution of **V** in THF. According to X-ray diffraction data, the unit cell of complex **V** contained one THF molecule. This molecule was lost during vacuum drying.

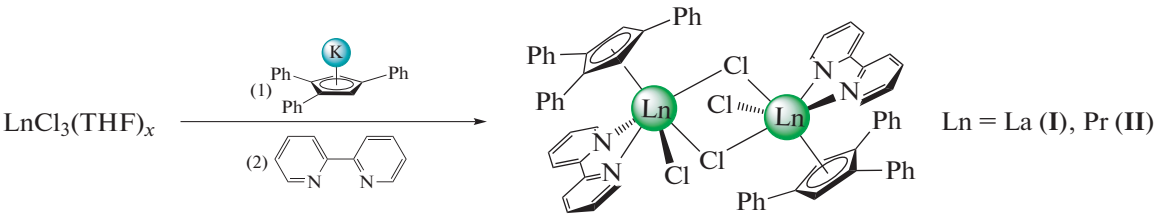
X-ray diffraction analysis of complexes **I**–**V** was carried out on a Bruker Quest D8 diffractometer (Photon-III detector, MoK_α radiation, ω-scan mode). The reflection intensities were determined using the SAINT program [13]. The absorption corrections were applied semiempirically based on equivalent reflections using the SADABS software [14]. The structures were solved by the direct method using the SHELXT program [15] and refined with the least-squares method in the full-matrix anisotropic approximation on F^2_{hkl} with the SHELXL-2018 software [16]. The disordered moieties were refined by applying constraints for atomic displacement parameters and positional parameters (DFIX and EADP). Analysis of the difference electron density maps and atomic displacement parameters revealed a pronounced disorder of the solvent (apparently, mainly THF) in the crystals of **I**, **II**, and **V**. The contribution of these disordered molecules was eliminated from the structural factors using the SQUEEZE program [17]. The hydrogen atoms in all structures were calculated using the riding model (C–H distances of 0.980 Å for methyl, 0.990 Å for methylene, and 1.000 Å for cyclopentadienyl hydrogen atoms) and refined in the relative isotropic approximation $U_{iso}(H) = 1.2U_{eq}(C)$. The main crystallographic data and refinement parameters for compounds **I**–**V** are summarized in Table 1.

The atom coordinates and other parameters of the structures of **I**–**V** were deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 2308607–2308611) deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

The reactions of REE chloride tetrahydrofuranates $LnCl_3(THF)_x$ (Ln = La, Pr, Er, Lu, Sc; THF = tetrahydrofuran) with triphenylcyclopentadienyl potassium and then with bipyridine in tetrahydrofuran affords heteroleptic complexes containing triphenylcyclopentadienyl and bipyridine ligands in the metal coordination sphere.

It is of interest that this type of ligand environment of REE ions, that is, triphenylcyclopentadienyl and bipyridine ligands in combination with two chloride ligands, gives rise to quite diverse geometry of the metal coordination sphere and, consequently, broad variety of structures of complexes with the same set of the ligands. In the case of lanthanum and praseodymium, binuclear complexes $[Cp^{Ph3}Ln(Bipy)Cl(\mu_2\text{-}Cl)]_2$ are formed (Ln = La (**I**), Pr(**II**); Scheme 1). Complexes **I** and **II** were obtained by recrystallization from 1 : 1 THF–hexane mixtures as orange-yellow (**I**) and yellow-green (**II**) crystals sparingly soluble in THF and insoluble in toluene.



Scheme 1. Synthesis of lanthanum and praseodymium triphenylcyclopentadienyl-bipyridine complexes.

The structures of **I** and **II** were determined by X-ray diffraction analysis (Fig. 1, Tables 1, 2). In the binuclear isostructural complexes **I** and **II**, the molecules of which are arranged on the inversion centers, the $[Cp^{Ph3}Ln(Bipy)Cl]$ moieties are connected by two bridging chloride ligands. Thus, each of the Ln^{3+} cations is coordinated to the triphenylcyclopentadienyl anion, two bridging and one terminal chloride ligands, and two bipyridine nitrogen atoms (the metal coordination number is 8). The ligands form a pseudo-octahedral environment around the metal, if the cyclopentadienyl ligand is considered to occupy one vertex of the octahedron. The $Ln–Cl_{term}$ distances are much shorter than the $Ln–Cl_{bridge}$ distances (see Table 2), and the $Cp_{centroid}LnCl_{term}$ angles in **I** and **II** are 105.4° and 104.7°, respectively.

Similar reactions of erbium, lutetium, and scandium tetrahydrofuranates with triphenylcyclopentadienyl potassium and bipyridine lead to the formation of mononuclear complexes $[Cp^{Ph3}Ln(Bipy)Cl_2(THF)]$ (Ln = Er (**III**), Lu (**IV**)) and $[Cp^{Ph3}Sc(Bipy)Cl_2]$ (**V**) (Scheme 2).

Table 1. Main crystallographic data and structure refinement details for compounds **I**–**V**

Parameter	Value				
	I	**II**	**III**	**IV**	**V**
Molecular formula	$C_{66}H_{50}N_4Cl_4La_2$, C_4H_8O	$C_{66}H_{50}N_4Cl_4Pr_2$, C_4H_8O	$C_{37}H_{33}N_2OCl_2Er$	$C_{37}H_{33}N_2OCl_2Lu$	$C_{33}H_{25}N_2OCl_2Sc$, C_4H_8O
M	1390.82	1394.79	759.81	767.52	637.51
T, K	110(2)	120(2)	100(2)	110(2)	110(2)
Crystal system	Monoclinic	Monoclinic	Orthorhombic	Orthorhombic	Monoclinic
Space group	$P2_1/n$	$P2_1/n$	$Pbca$	$Pbca$	$P2_1/c$
Z (Z')	2 (1)	2 (1)	8 (1)	8 (1)	4 (1)
a, Å	17.852(2)	17.794(4)	16.4730(5)	16.4684(14)	14.746(3)
b, Å	10.1669(16)	10.134(2)	16.5182(5)	16.4320(18)	14.701(3)
c, Å	18.544(3)	18.503(4)	23.365(1)	23.306(2)	15.749(3)
β, deg	109.348(4)	108.982(7)	90	90	109.49(3)
V, Å^3	3175.6(8)	3155.0(12)	6357.7(4)	16.4684(14)	3218.4(13)
ρ(calcd.), g cm^{-3}	1.445	1.454	1.588	1.617	0.427
μ, mm^{-1}	1.541	1.735	2.840	3.333	0.689
F(000)	1392	1400	3032	3056	1328
$2\theta_{max}$, deg (completeness)	52 (0.998)	54	58 (1.000)	48 (0.994)	58 (0.993)
Number of measured reflections	21938	16280	46953	47223	22946
Number of unique reflections	6230	6800	6258	6208	8494
Number of reflections with $I > 2\sigma(I)$	4416	3791	5019	4291	4514
Number of refined parameters	202	344	388	388	343
R_1 ($I > 2\sigma(I)$)	0.0678	0.0863	0.0338	0.0393	0.0597
wR_2 (all data)	0.1668	0.2242	0.0843	0.0894	0.1596
GOOF	1.049	1.001	0.929	1.019	0.939
Residual electron density (min/max), e Å^{-3}	−1.745/1.882	−1.445/1.465	−0.719/0.984	−1.037/0.573	−0.354/0.342

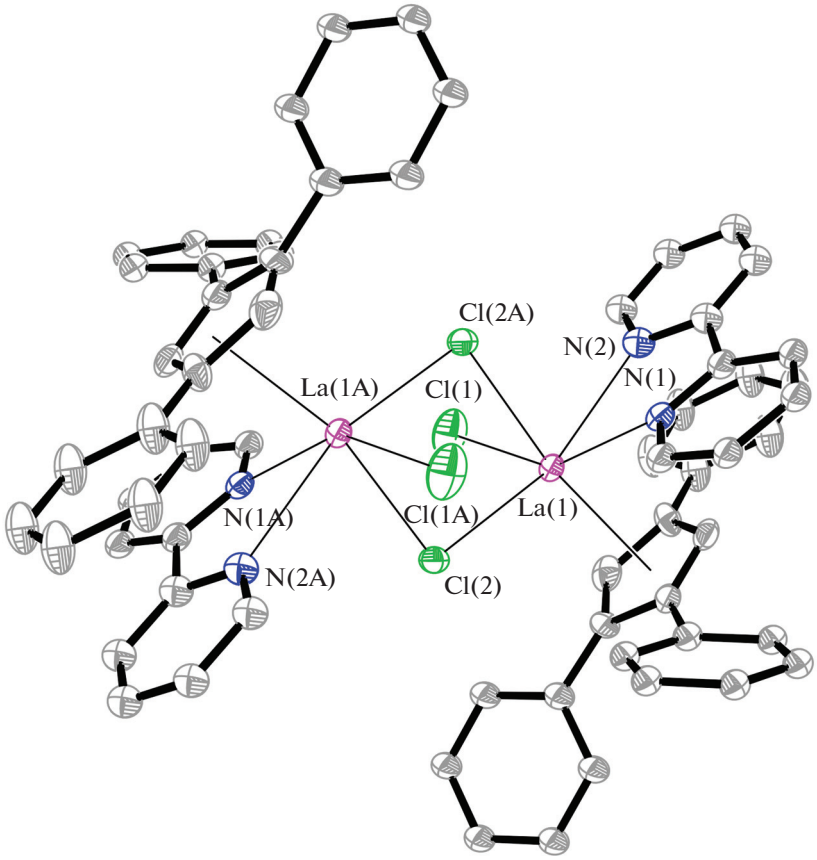
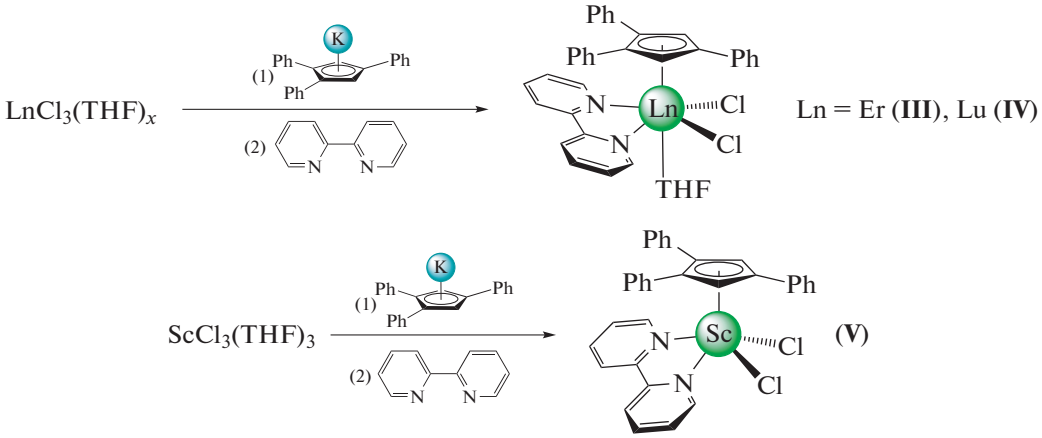


Fig. 1. Structure of complex **I** with atoms represented by thermal ellipsoids (ρ = 50%). Hereinafter, hydrogen atoms and phenyl substituents are not shown for the sake of simplicity.



Scheme 2. Synthesis of complexes **III**–**V**.

Unlike lanthanum and praseodymium, the ions of which have relatively large ionic radii, the other 4*f* elements give mononuclear complexes [Cp^{Ph3}Ln(Bipy)Cl_2(THF)] (**III**, **IV**) (C.N. = 8) (Ln = Er, Lu), which we described previously [8], similar to Nd, Gd, and Tb tri- and tetraphenylcyclopentadienyl complexes which we described previously [8]. In the case of scandium, which has the smallest ionic radius among REEs, the complex [Cp^{Ph3}Ln(Bipy)Cl_2] (**V**) is formed (C.N. = 7). Like the previously described complexes, the obtained compounds have stacking interactions between the phenyl substituents of the cyclopentadienyl rings and bipyridine ligands. Characteristic features of these interactions are discussed below.

Table 2. Selected structural parameters of the triphenylcyclopentadienyl-bipyridine complexes

Complex	Ln–Cl bond lengths, Å	Ln–N bond lengths, Å	Ln–O bond length, Å	Ln–$Cp_{centroid}$ distance, Å	Rotation angles of the phenyl ring in position 4, deg	Rotation angles of the phenyl rings in positions 1 and 2, deg	NLnN angle, deg
I	2.739(3) (term.); 2.928(2), 2.830(2) (bridge)	2.59(1)–2.69(1)		2.60	5.9	41.4, 34.0	58.6(4)–62.3(4)
II	2.689(3) (term.); 2.793(3), 2.886(3) (bridge)	2.59(1)		2.542	12.0	34.0, 38.1	62.6(3)
III	2.5715(9), 2.5774(9)	2.451(3)–456(3)	2.452(2)	2.409	7.2	22.1, 50.5	66.8(1)
IV	2.539(1), 2.546(1)	2.411–2.412 (5)	2.437(4)	2.384	6.3	21.8, 51.5	67.92(15)
V	2.315(1), 2.324 (1)	2.315(3)–2.324(2)		2.209	6.8	43.3, 39.9	69.7(1)

Complexes **III** and **IV** are isostructural, the lanthanide cation in them being coordinated to the cyclopentadienyl anion, two chloride ligands, two bipyridine nitrogen atoms, and the THF oxygen atom (Fig. 2). The primary difference between **III** or **IV** and the complexes of lighter lanthanides is the geometry of

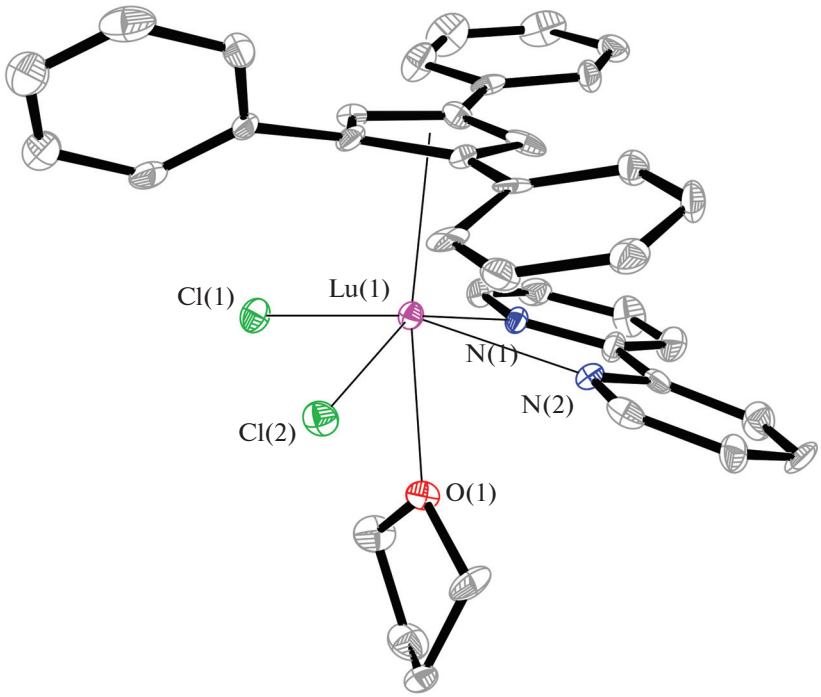


Fig. 2. Structure of complex **IV** with atoms represented by thermal ellipsoids (ρ = 50%).

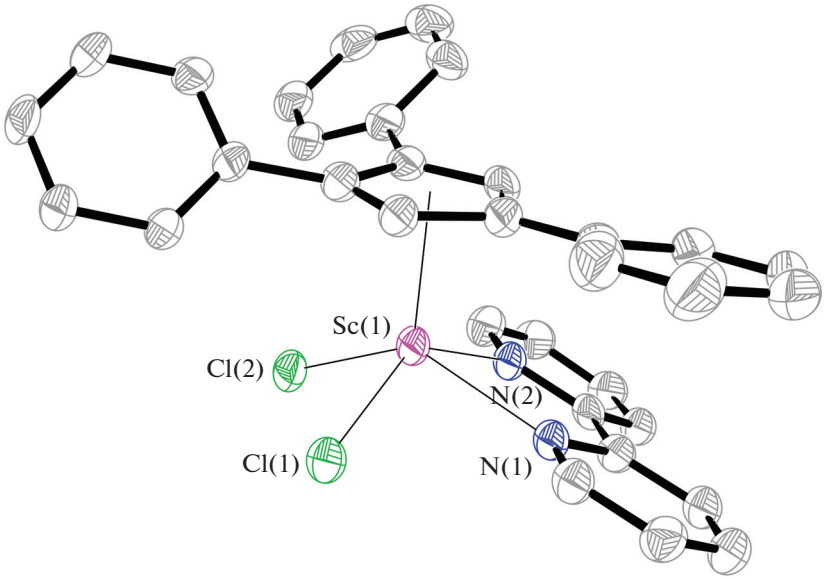


Fig. 3. Structure of complex **V** with atoms represented by thermal ellipsoids (ρ = 50%).

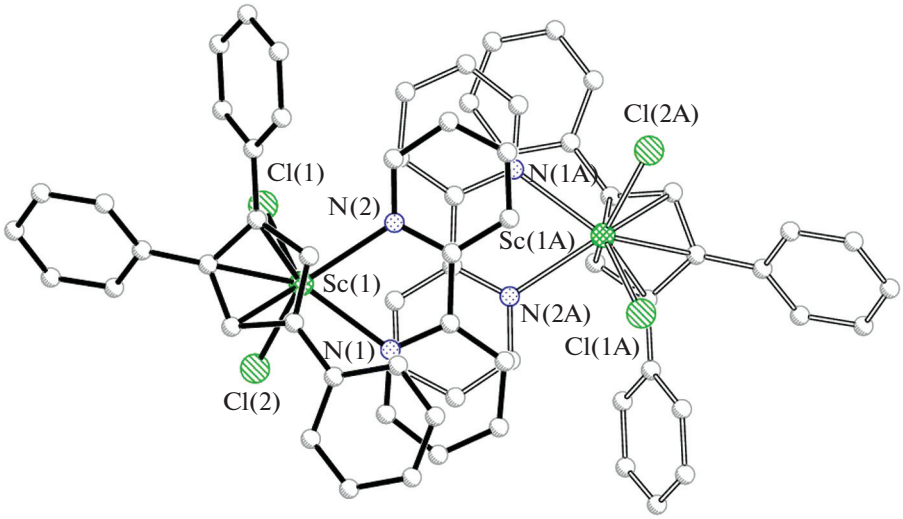


Fig. 4. Centrosymmetric stacking dimers in the crystal structure of **V**.

the bipyridine ligand: the lanthanide atom almost does not deviate from the bipyridine ligand plane.

Complex **V** differs from complexes **III** and **IV** by the absence of the metal-coordinated tetrahydrofuran molecule, the Sc C.N. being 7 (Fig. 3). The metal coordination polyhedron can be described as a distorted tetragonal pyramid with the cyclopentadienyl ligand at a vertex. The distance from scandium to the ClClNN plane is 0.896 Å. The absence of a solvated THF molecule in the coordination sphere, which shields the bipyridine π-system, leads to a somewhat different intermolecular packing. Whereas in all compounds considered above, the intermolecular interactions are mainly H...H and C...H contacts, in the case of **V**, stacking interaction with the C...C distance of 3.45 Å is implemented (Fig. 4).

As we noted above, the presence of intramolecular stacking interaction can be assumed for the crystals of **I**–**V** in all cases (Fig. 5). This is attributable to the fact that the chloride anions and nitrogen atoms in these complexes are located approximately in the same plane (the standard deviation is not more than 0.05 Å). This plane is coplanar to the cyclopentadienyl ligand, which, enables the overlap of the π-systems of the phe-

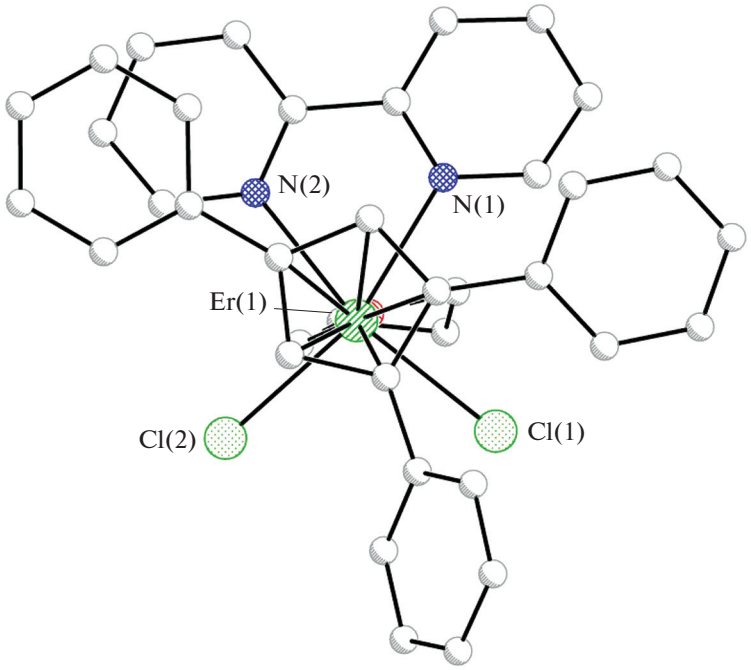


Fig. 5. Character of overlap of the π-systems involved in the intramolecular stacking interactions.

nyl substituents and the bipyridine ligand. In view of the fact that the phenyl groups in positions 1 and 2 are always rotated relative to the cyclopentadienyl ligand, the only possible pair of rings to form the stacking interactions are the phenyl group in position 4 of cyclopentadienyl and bipyridine. The efficiency of the stacking interaction is also controlled by the ionic radius of the rare earth element ion.

It is noteworthy that in the three different types of complexes (as regards the crystal symmetry), the type of overlap of the π-system actually does not change, with the angles between the phenyl group in position 4 and the pyridine ring being 8.6° in complexes **I** and **II**, 17° in **III** and **IV**, and 19° in **V**. The shortest distances between carbon atoms of different aromatic rings are in the range of 3.1–3.3 Å.

In this study, we obtained and structurally characterized mono- and binuclear heteroleptic REE triphenylcyclopentadienyl bipyridine complexes, commonly characterized by the coplanar arrangement of two different π-systems (triphenylcyclopentadienyl ligand and bipyridine), which may implement stacking interaction. Interestingly, this pattern holds for both mononuclear and binuclear complexes. Since the indicated π-systems can act as π-type (triphenylcyclopentadienyl) and σ-type (bipyridine) antenna ligands, this opens up the possibility of preparing also heterometallic complexes promising as regards the photophysical properties using the same set of ligands.

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CONFLICT OF INTEREST

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

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