

Dedicated to M.N. Bogachev in celebration of his 85th birthday

Trinuclear Lutetium(III) Cyclopentadienyl Complex with the 2,2'-Bipyridine Dianion

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Received December 5, 2023; revised January 19, 2024; accepted January 25, 2024

Abstract—The reaction of lutetium cyclopentadienyl anthracenide complex $[(C_5H_5)Lu(C_{14}H_{10})(THF)_2]$ with 1 equiv. of 2,2'-bipyridine in THF gives the trinuclear complex $[(\eta^5-C_5H_5)Lu]_3(\mu_2-Bipy)_3$ (**I**), containing a 2,2'-bipyridine dianion. The complex was isolated as a powder with the composition $I \cdot 0.1(C_{14}H_{10}) \cdot 0.8(C_7H_8)$. The recrystallization from a toluene/hexane mixture resulted in the crystals of $I \cdot 0.084(C_{14}H_{10}) \cdot 0.831(C_7H_8) \cdot 0.500(C_6H_{14})$, which were studied by X-ray diffraction (monoclinic group $P2_1/c$; CCDC no. 2311508). Complex **I** has an unusual $\mu_2-\kappa^2N^1, N^1':\eta^4N^1, C^2, C^2', N^1'$ -bridging coordination of the dianion.

Keywords: rare earth elements, lutetium, 2,2-bipyridine, dianion, X-ray diffraction analysis

DOI: 10.1134/S1070328424600165

INTRODUCTION

It is known that $4n \pi$ -electron dianions of aromatic hydrocarbons and heterocyclic analogues (L^{2-}) fully comply with the generally accepted criteria for the formation of kinetically stable organometallic complexes of rare earth elements (REEs) [1]. There are known REE complexes with dianions derived from pyrene, benzantracene, anthracene, naphthalene, benzene [2], biphenyl [3], 2,3-dimethylquinoxaline, phenazine [4], 2,2'-bipyridine (Bipy) [5–7], and its substituted derivatives [8, 9]. Currently, studies of the dianion rare earth metal complexes are mainly focused on the complexes like $[Ln^{3+}X_2^-]_2[\mu-L^{2-}](X^- \text{ is a monoanionic ligand})$, which can be considered as ionic triples in which the L^{2-} dianion is coordinated in the bridging mode and formally acts as two separate monoanions. However, of most interest are poorly investigated complexes like $[Ln^{2+}L^{2-}]_n$, $[X-Ln^{3+}L^{2-}]$, $[X_2^-Ln^{3+}L^{2-}]^-$, and $[Ln^{3+}L_2^{2-}]^{2-}$, in which the interaction between highly polarizing $Ln^{2+/3+}$ cation and easily polarizable L^{2-} dianion can be implemented in full measure. In complexes of this type, the L^{2-} ligand is often characterized by a substantial HOMO localization, which, for exam-

ple, may result in an essentially covalent character of the $Ln-C$ bond [10, 11]. Among the complexes with dianions derived from the heteroatomic analogues of aromatic hydrocarbons, structures of REE complexes with the 2,2'-bipyridine ($Bipy^{2-}$) dianion have been least studied. Apparently, this is attributable to the difficulty of preparation and isolation of these complexes, caused by a high reducing ability of $Bipy^{2-}$.

On the other hand, the structures of REE complexes with the $Bipy^{\bullet-}$ radical anion (or those with simultaneously two ligands, formally $Bipy^{\bullet-}$ and $Bipy^0$) have been studied more extensively: according to the Cambridge Crystallographic Data Centre (CCDC, version 2022.3.0) [12, 13], the structures of 25 such complexes are known [6, 14–25]. In these complexes, the $Bipy^{\bullet-}$ ligand is most often planar or nearly planar and the $C_{ipso}-C'_{ipso}$ bond length tends to decrease (on average, 1.42 Å) in comparison with that in REE complexes with $Bipy^0$ (more than 650 structures; the average $C_{ipso}-C'_{ipso}$ bond length in REE complexes is 1.48 Å) or with uncoordinated $Bipy^0$ (1.49 Å).

The REE complexes with Bipy²⁻ ligands (5 structures) or with Bipy²⁻ derivatives (5 structures) can be prepared by several methods: (1) reduction of Bipy⁰ or Bipy^{•-} directly in the metal coordination sphere ([Y(Tp^{Me2})(Bipy)(THF)₂] [6], [Cp₂^{*}Ln(Bipy)]⁻ (Ln = Nd, Sm, Gd) [7], [Cp₂^{*}Ln{4,4'-(Mes₂B)₂-2,2'-Bipy}]⁻ (Ln = Dy, Gd, [26]), (2) the exchange reaction of REE halide with alkali metal bipyridine complex ([Yb(μ₂-Bipy)(THF)₂]₃ [5]), (3) the exchange redox reaction of Bipy⁰ with REE complexes containing a dianion (L²⁻) that is a more potent reducing agent than Bipy²⁻ ([Yb(μ₂-Bipy)(THF)₂]₃ [5]), and (4) the reaction of REE alkyl complex, [(ArNCH₂CH₂.NAr)Y(THF)₂(CH₂TMS)], with 2-aryl-substituted pyridine ([[(ArNCH₂CH₂NAr)-(THF)Y]₂[μ₂-3,3'-Ar'₂-2,2'-Bipy] (Ar' = Ph, *p*-Tol, 4-MeOC₆H₄) [27]). In all known REE complexes with Bipy²⁻, the C_{ipso}-C_{ipso} bond is shortened to 1.41–1.35 Å.

In the present study, we examined the possibility of preparing complexes [Cp⁻Ln³⁺(Bipy)²⁻] by exchange reaction (3) starting from the anthracenide complex [Cp⁻Lu³⁺(C₁₄H₁₀)²⁻(THF)₂] and investigated the types of coordination of the 2,2'-bipyridine dianion in the [Cp⁻Ln³⁺(Bipy)²⁻] complexes.

EXPERIMENTAL

Synthetic operations were carried out in the atmosphere of purified argon in anhydrous solvents using a SPECS-GB2 glove box. Tetrahydrofuran was pre-dried over NaOH and distilled from potassium/benzophenone. Hexane was distilled from sodium–potassium eutectic/benzophenone. Toluene was distilled from potassium/benzophenone. LuCl₃(THF)₃ [28], [CpLuCl₂(THF)₃] [29], and [CpLu(C₁₄H₁₀)(THF)₂] [30] (THF = tetrahydrofuran, Cp = C₅H₅) were prepared by known procedures. 2,2'-Bipyridine and anthracene were purified prior to use by sublimation in a dynamic vacuum of 5 × 10⁻² mmHg. THF-*d*₈ (Sigma-Aldrich, 99.5 at % D) was distilled from sodium–potassium eutectic, stored over sodium–potassium eutectic and anthracene, and condensed into NMR tubes. The ¹H, ¹³C{¹H}, ¹H–¹H COSY, ¹³C–¹H HSQC, and ¹H DOSY NMR spectra were recorded on a Bruker AVANCE III HD instrument (operation frequency of 400 MHz for ¹H). Elemental analysis was carried out on a Thermo Scientific FLASH 2000 CHNS/O Analyzer instrument.

Synthesis of [CpLu(Bipy)]₃(C₁₄H₁₀)_{0.1}(C₇H₈)_{0.8} (I·0.1(C₁₄H₁₀)·0.8(C₇H₈)). A solution of Bipy (0.024 g, 0.154 mmol) in THF (3 mL) was slowly added with stirring to a solution of [CpLu(C₁₄H₁₀)(THF)₂] (0.086 g, 0.153 mmol) in

THF (5 mL). The color of the reaction mixture changed from dark red to dark brown. The reaction mixture was stirred for 30 min, centrifuged (4000 rpm, 5 min), and evaporated to dryness under dynamic vacuum. Toluene (8 mL) was added to the solid residue, and the mixture was centrifuged (4000 rpm, 5 min). Hexane (20 mL) was carefully added to the resulting solution, so as to avoid mixing of the layers. After several weeks, dark brown, nearly black, crystals were formed. The crystals were dried under dynamic vacuum to a constant weight to give 0.042 g (0.033 mmol, 64%) of complex I·0.1(C₁₄H₁₀)·0.8(C₇H₈). The composition was determined on the basis of ¹H NMR spectroscopy and elemental analysis data.

For C₅₂H_{46.4}N₆Lu₃

Anal. calcd., %	C, 48.76	H, 3.65	N, 6.56
Found, %	C, 47.71	H, 3.18	N, 6.68

¹H NMR (THF-*d*₈; δ, ppm): 2.31 (s, 2.5H, CH₃, toluene), 5.01 (t, 6H, ³J_{H–H} = 6.3 Hz, CH, bipyridine), 5.94 (dd, 6H, ³J_{H–H} = 9.7, 5.7 Hz, CH, bipyridine), 6.00 (s, 15H, Cp-*H*), 6.50 (d, 6H, ³J_{H–H} = 9.7 Hz, CH, bipyridine), 6.56 (d, 6H, ³J_{H–H} = 6.7 Hz, CH, bipyridine), 7.05–7.22 (m, 4.5H, C₆H₅, toluene), 7.43 (m, 0.4H, C_{2,3,6,7}-*H*, anthracene), 8.00 (m, 0.4H, C_{1,4,5,8}-*H*, anthracene), 8.45 (s, 0.2H, C_{9,10}-*H*, anthracene). ¹³C{¹H} NMR (THF-*d*₈; δ, ppm): 21.6 (CH₃, toluene), 103.8 (CH, bipyridine), 111.3 (Cp–C), 120.1 (C_{ipso}, bipyridine), 120.8 (CH, bipyridine), 123.3 (CH, bipyridine), 126.2 (CH_{para}, toluene), 129.1 (CH_{meta}, toluene), 129.6 (CH_{ortho}, toluene), 141.4 (CH, bipyridine).

The crystals of I·0.084(C₁₄H₁₀)·0.831(C₇H₈)·0.500(C₆H₁₄) suitable for X-ray diffraction were obtained by slow diffusion of hexane into a solution of I in toluene. According to X-ray diffraction data, the unit cell contained hexane molecules, which were lost upon vacuum drying of complex I. Recrystallization under indicated conditions did not result in complete removal of anthracene molecules from the crystal.

X-ray diffraction analysis of the crystals of I·0.084(C₁₄H₁₀)·0.831(C₇H₈)·0.500(C₆H₁₄) was carried out on a Rigaku XtaLAB Synergy-S four-circle diffractometer (HyPix6000HE detector, κ-geometry, shutterless ω-scan mode, PhotonJet-S microfocus sealed tube, monochromatization by a system of mirrors, MoK_α radiation, λ = 0.71073 Å). The reflection intensities were collected and analytically corrected for absorption by the crystal using the CrysAlisPro program [31]. The structure was solved by the direct method with the SHELXT program [32] and refined with the least-squares methods in the full-matrix anisotropic approximation on *F*_{hkl}² using the Olex2 software package [33] and the SHELXL-2018 pro-

Table 1. Main crystallographic data and structure refinement details for the crystal of $\text{I} \cdot 0.084(\text{C}_{14}\text{H}_{10}) \cdot 0.831(\text{C}_7\text{H}_8) \cdot 0.500(\text{C}_6\text{H}_{14})$

Parameter	Value
Molecular formula	$\text{C}_{55}\text{H}_{53.49}\text{N}_6\text{Lu}_3$
M	1323.44
Temperature, K	99.9(3)
System	Monoclinic
Space group	$P2_1/c$
a , Å	12.09591(7)
b , Å	18.07628(11)
c , Å	21.48856(13)
β , deg	101.1297(6)
V , Å ³	4610.09(5)
Z/Z'	4/1
ρ (calcd.), g cm ⁻³	1.907
μ , mm ⁻¹	6.419
$F(000)$	2554
Crystal size, mm	$0.16 \times 0.14 \times 0.10$
Color	Dark violet
Habit	Block
Data collection range over θ , deg	2.053–32.499
Ranges of hkl indices	$-18 \leq h \leq 18, -27 \leq k \leq 27, -32 \leq l \leq 32$
Number of reflections: collected	156 136
unique (R_{int})	16681 (0.0306)
observed with $I > 2\sigma(I)$	15479
Completeness to θ_{max}	1.000
$T_{\text{max}}/T_{\text{min}}$	0.602/0.466
Data/constraints/parameters	16681/8/757
Parameter S (on F^2)	1.084
R_1/wR_2^* (for reflections with $I > 2\sigma(I)$)	0.0169/0.0399
R_1/wR_2^* (for all data)	0.0198/0.0408
$\Delta\rho_{\text{max}}/\Delta\rho_{\text{min}}$, e Å ⁻³	1.777/–1.160

* $R_1 = \sum \|F_o\| - |F_c| / \sum \|F_o\|$, $wR_2 = [\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}$.

gram [34]. The hydrogen atom positions in the complex were found and refined using difference electron density maps in the isotropic approximation. The anthracene, hexane, and toluene hydrogen atoms were placed in ideal calculated positions (C–H distances of 0.950 Å for aromatic, 0.980 Å for methyl, and 0.990 Å for methylene hydrogen atoms) and refined as riding atoms in the relative isotropic approximation $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for other hydrogen atoms. The rotating methyl group model was used. Since the toluene and

anthracene molecules had a partial site occupancy and were located at the same place (see Results and Discussion), this disorder was modeled applying constraints for atomic displacement parameters and positional parameters (SADI and EADP instructions of the SHELXL software). The main crystallographic data and refinement parameters for compound $\text{I} \cdot 0.084(\text{C}_{14}\text{H}_{10}) \cdot 0.831(\text{C}_7\text{H}_8) \cdot 0.500(\text{C}_6\text{H}_{14})$ are summarized in Table 1.

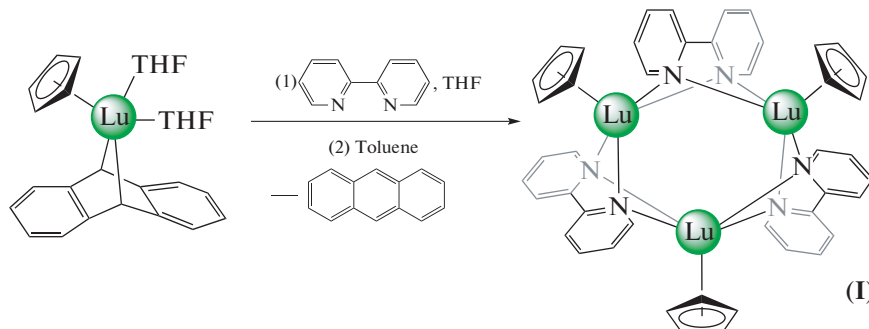
The atom coordinates and other parameters of the structure of $\text{I} \cdot 0.084(\text{C}_{14}\text{H}_{10}) \cdot 0.831(\text{C}_7\text{H}_8) \cdot 0.500(\text{C}_6\text{H}_{14})$

were deposited with the Cambridge Crystallographic Data Centre (CCDC no. 2311508; deposit@ccdc.cam.ac.uk or <https://www.ccdc.cam.ac.uk/structures>).

RESULTS AND DISCUSSION

The addition of bipyridine to the lutetium cyclopentadienyl anthracenide complex $[\text{CpLu}(\text{C}_{14}\text{H}_{10})\text{-(THF)}_2]$ in THF results in immediate color change from characteristic dark red to dark brown (Scheme 1). Evaporation of THF from the reaction

mixture in vacuo gives a powder containing the complex $[\text{CpLu}(\text{Bipy})]_3$ (**I**). The powder recrystallization from a toluene/hexane mixture yields microcrystals of compound $\text{I} \cdot 0.1(\text{C}_{14}\text{H}_{10}) \cdot 0.8(\text{C}_7\text{H}_8)$ (^1H NMR data), the composition of which was confirmed by elemental analysis. The isolated powder had a moderate solubility in THF and toluene, but was insoluble in hexane. The attempts to get rid of anthracene present in the co-crystallize $\text{I} \cdot 0.1(\text{C}_{14}\text{H}_{10}) \cdot 0.8(\text{C}_7\text{H}_8)$ by reprecipitation or recrystallization failed.



Scheme 1. Synthesis of $[\{(\eta^5\text{-C}_5\text{H}_5)\text{Lu}\}_3(\mu_2\text{-Bipy})_3]$.

The ^1H NMR spectrum of complex $\text{I} \cdot 0.1(\text{C}_{14}\text{H}_{10}) \cdot 0.8(\text{C}_7\text{H}_8)$ in $\text{THF-}d_8$ exhibits, along with toluene and anthracene signals, also a singlet for the protons of the cyclopentadienyl anion and four signals of equal intensity in the region of 5.01–6.56 ppm, which correspond to protons of the coordinated bipyridine dianion. The upfield shift of Bipy^{2-} proton signals with respect to Bipy^0 proton signals (7.32–8.70 ppm in CDCl_3 [35]) confirms the assumption about the dianionic nature of the ligand in complex **I**.

The recrystallization of powdered $\text{I} \cdot 0.1(\text{C}_{14}\text{H}_{10}) \cdot 0.8(\text{C}_7\text{H}_8)$ from a toluene/hexane mixture resulted in the formation of crystals of $\text{I} \cdot 0.084(\text{C}_{14}\text{H}_{10}) \cdot 0.831(\text{C}_7\text{H}_8) \cdot 0.500(\text{C}_6\text{H}_{14})$ suitable for X-ray diffraction (Table 1). The crystallographically independent part of the unit cell includes complex **I** and organic molecules (Fig. 1). The unit cell contains a toluene molecule (C(49)...C(55) atoms), located near the inversion center, with a probability of 0.831(2). In other cases, the unit cell contains an anthracene molecule located at the same inversion center, with the site occupancy of the C(60)...C(66) atoms being 0.169(2). The hexane molecule is also located at the inversion center. This arrangement of molecules corresponds to their following ratio in the crystal: $0.084(\text{C}_{14}\text{H}_{10}) : 0.831(\text{C}_7\text{H}_8) : 0.500(\text{C}_6\text{H}_{14})$ per molecule of **I**.

In trinuclear complex **I**, each cyclopentadienide anion is symmetrically η^5 -coordinated by one Lu^{3+} cation, since the $\text{Lu-Cp}(\text{centroid})$ and $\text{Lu-Cp}(\text{plane})$ distances are equal (Table 2). The average

C–C and C–N bond lengths for the coordinated Bipy^0 , $\text{Bipy}^{\cdot-}$, and Bipy^{2-} ligands were analyzed in the literature [5, 6], while the most characteristic distance is the $\text{C}_{\text{ipso}}\text{--C}'_{\text{ipso}}$, which ranges from 1.35(2) Å for the mononuclear $[\text{Y}(\text{Tp}^{\text{Me}_2})(\text{Bipy})(\text{THF})_2]$ complex (non-planar Bipy^{2-}) [6] to 1.41 Å for $[\text{Yb}(\mu_2\text{-Bipy})(\text{THF})_2]_3$ (planar Bipy^{2-}) [5]. In complex **I**, this distance corresponds to the middle of this range: 1.382(2)–1.389(3) Å (Table 2). The Bipy^{2-} dianion in **I** has an unusual $\mu_2\text{-}\kappa^2\text{N}^1, \text{N}^{1'} : \eta^4\text{N}^1, \text{C}^2, \text{C}^{2'}, \text{N}^{1'}$ -bridging coordination to Lu^{3+} , found in only one complex: $[\text{Yb}(\mu_2\text{-Bipy})(\text{THF})_2]_3$ [5]. The dianion in **I** is non-planar, unlike that in $\text{Yb}(\text{II})$ complex: the dihedral angles between the pyridine planes (C_5N) are in the $12.4^\circ\text{--}16.2^\circ$ range, and the C_{ipso} atom deviates from the plane of the neighboring pyridine moiety by 0.17–0.28 Å, with the $\text{N}(1)\text{C}(2)\text{C}(2')\text{N}(1')$ torsion angles in the dianion being only $0.3^\circ\text{--}2.3^\circ$. Considering the rotation of the Cp rings and the conformation of the dianion, complex **I** has local symmetry C_3 , but it is located in a general position in the unit cell. The average Lu-Lu distance (3.60 Å) in **I** is 0.09 Å shorter than the average Yb-Yb distance (3.69 Å). Despite the greater steric crowding of **I** and smaller radius of the Lu^{3+} cation compared to Yb^{2+} (the Lu^{3+} coordination number is greater by unity; $r(\text{Yb}^{2+})\text{--}r(\text{Lu}^{3+}) = 0.10$ Å [36]), the Lu^{3+} cation forms the same structural motif: $[\text{Ln}(\mu_2\text{-}\kappa^2\text{-}\eta^4\text{-Bipy})]_3$.

The ^1H DOSY NMR study made it possible to estimate the average hydrodynamic radius of complex **I** in

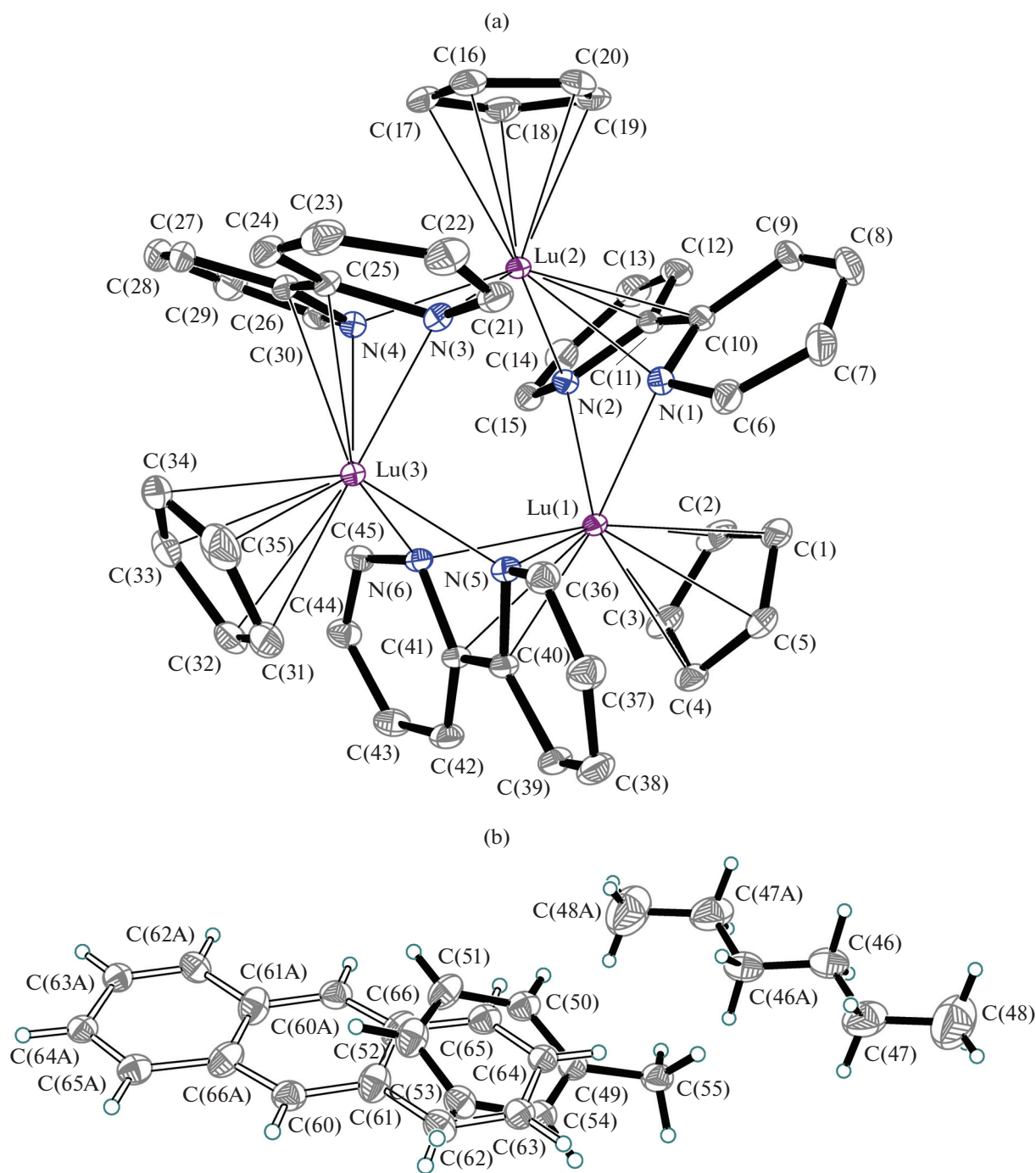


Fig. 1. Molecular structure of $\{[(\eta^5\text{-C}_5\text{H}_5)\text{Lu}]_3[\mu_2\text{-}\kappa^2\text{N}^1, \text{N}^{1'}:\eta^4\text{N}^1, \text{C}^2, \text{C}^{2'}, \text{N}^{1'}\text{-Bipy}]_3\}$ (**I**): (a) atoms (hydrogen atoms are omitted) and (b) non-coordinating organic molecules (the minor disorder component, anthracene, is shown with transparent lines) for $\text{I} \cdot 0.084(\text{C}_{14}\text{H}_{10}) \cdot 0.831(\text{C}_7\text{H}_8) \cdot 0.500(\text{C}_6\text{H}_{14})$ ($p = 50\%$).

a THF solution ($r_s = 7.5 \pm 0.3$ Å). However, such an estimate without direct measurement of the diffusion coefficient and dynamic viscosity or without using an internal standard tends to overestimate r_s for similar systems in THF- d_8 . The calculation of the Connolly surface (solvent accessible surface area) based on

X-ray diffraction data for trinuclear complex **I** with a probe radius of 2.6 Å characteristic of THF [37] gives $r_s = 6.9$ Å. Thus, on the basis of DOSY studies, it can be assumed that complex **I** in THF is oligomeric.

Thus, in this study, we prepared for the first time a Ln(III) complex containing Cp^- and Bipy^{2-} ligands

Table 2. Selected bond lengths and distances (Å) in **I**

Bond	Value		
	Lu(1)	Lu(2)	Lu(3)
Lu–N _{Bipy} (κ^2)	2.3361(14), N(1)	2.3415(14), N(3)	2.3426(14), N(5)
	2.3439(14), N(2)	2.3516(14), N(4)	2.3365(14), N(6)
Lu–N _{Bipy} (η^4)	2.4499(14), N(5)	2.4466(14), N(1)	2.4282(14), N(3)
	2.4365(14), N(6)	2.4155(14), N(2)	2.4240(14), N(4)
Lu–C _{Bipy} (η^4)	2.5647(16), C(40)	2.5666(16), C(10)	2.5655(16), C(25)
	2.5648(16), C(41)	2.5691(16), C(11)	2.5825(15), C(26)
Cp atoms	C(1)...C(5)	C(16)...C(20)	C(31)...C(35)
Lu–C _{Cp} (aver.)	2.5834(18)	2.5851(19)	2.5910(18)
C _{Cp} –C _{Cp} (aver.)	1.411(3)	1.410(3)	1.409(3)
Lu–C _{Cp} (centroid)	2.2878(8)	2.2901(8)	2.2976(8)
Lu–C _{Cp} (plane)	2.2877(8)	2.2900(8)	2.2976(8)
C _{ipso} –C _{ipso}	1.389(3), C(10)–C(11)	1.386(3), C(25)–C(26)	1.382(2), C(40)–C(41)
Lu...Lu	3.59948(9), Lu(1)...Lu(2)	3.60844(9), Lu(1)...Lu(3)	3.60167(9), Lu(2)...Lu(3)

using the ligand exchange reaction between [CpLu(C₁₄H₁₀)(THF)₂] and Bipy⁰. The molecular structure of the complex [(η^5 -C₅H₅)Lu]₃(μ_2 - κ^2 : η^4 -Bipy)₃] in the crystal was determined.

ACKNOWLEDGMENTS

The authors are grateful to A.V. Kiselev for the help in recording the NMR spectra using the research equipment of the Center for Collective Use “Analytical Center for Extensive Oil Refining and Petrochemistry Problems” of the Topchiev Institute of Petrochemical Synthesis, Russian Academy of Sciences.

FUNDING

This study was supported by the Russian Science Foundation (grant no. 22-23-00711).

CONFLICT OF INTEREST

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

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Translated by Z. Svitanko

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