ISSN 1070-3284, Russian Journal of Coordination Chemistry, 2015, Vol. 41, No. 1, pp. 51–57. © Pleiades Publishing, Ltd., 2015.
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Synthesis and Crystal Structure of the New Binuclear Complex of Cerium(III) Nitrate with 4,4,10,10-Tetramethyl-1,3,7,9-Tetraazospiro[5.5]undecane-2,8-Dione and the Change in the Periodical Properties in Isostructural Series of Lanthanide Complexes with This Ligand

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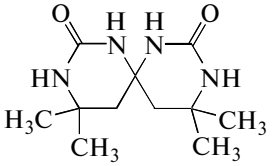
Received June 30, 2014

Abstract—The centrosymmetrical binuclar complex of cerium(III) nitrate with bicyclic bis-urea 4,4,10,10-tetramethyl-1,3,7,9-tetraazospiro[5.5]undecane-2,8-dione or spirocarbone (Sk), $[Ce(C_{11}H_{20}N_4O_2)(H_2O)_2(NO_3)_3]_2$ (**I**), is synthesized for the first time, and its structure is determined (CIF file CCDC 985758). The crystals of compound **I** are monoclinic: space group $P2_1/n$, a = 14.2797(13), b = 7.3922(6), c = 21.003(2) Å, β = 102.103(10)°, V = 2167.8(3) Å^3, ρ_{calcd} = 1.85 g/cm^3, Z = 2. The cerium atom is coordinated by two oxygen atoms of two Sk molecules bound by the symmetry procedure $(1 - x, -y, 1 - z)$, three bidentate nitrate anions, and two water molecules. The coordination polyhedron of the cerium atom is an irregular ten-vertex polyhedron. The Ce...Ce distance in the complex is 9.57 Å. The change in the periodical properties in a series of the Sk complexes with lanthanide(III) (Ce–Lu, except for Pm) nitrates is discussed.

DOI: 10.1134/S1070328415010066

INTRODUCTION

Coordination compounds of lanthanides with ligands of the class of cyclic spirobisureas are presently almost unstudied. One of these ligands is 4,4,10,10-tetramethyl-1,3,7,9-tetraazospiro[5.5]undecane-2,8-dione or spirocarbone ($C_{11}H_{20}N_4O_2$, Sk).



This ligand (precursor of urea) has a number of valuable biological properties: they are lowly toxic (LD_{50} = 3000 mg/kg of white mice weight) [1] and membranotropic [2] and can penetrate and be accumulated in the cytoplasm of leucosis mice and human line cells L1210 and CEM-T4, respectively [3]. The ligand favors an increase in the amount of protein and a decrease in starchiness in oats grains [4]. The efficiency of using spirocarbone as a stimulator for callus formation in Forsythia europaea and root formation in Snowbelle was proved [5]. The efficiency of using spirocarbone as a stimulator of growth and development in sheep breeding was shown [6]. Therefore, the synthesis and study of the coordination properties of this ligand as a hard Lewis base will clarify more completely the chemical nature of the interaction of Sk with atoms of various rare-earth metals.

The following binuclear spirocarbone complexes with rare-earth metals were synthesized and characterized: $[Y(C_{11}H_{20}N_4O_2)(H_2O)_3(NO_3)_2]_2(NO_3)_2$ (CCDC 903389) [7], $[La(C_{11}H_{20}N_4O_2)(H_2O)_2(NO_3)_3]_2$ (CCDC 903388) [8], $[Pr(C_{11}H_{20}N_4O_2)(H_2O)_3(NO_3)_2]_2(NO_3)_2$ (CCDC 924475) [9], $[Nd(C_{11}H_{20}N_4O_2)(H_2O)_3(NO_3)_2]_2(NO_3)_2$ (CCDC 876569) [10], $[Sm(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$ (CCDC 924470), $[Eu(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$ (CCDC 924469), $[Gd(C_{11}H_{20}N_4O_2)(H_2O)_3(NO_3)_2]_2(NO_3)_2$ (CCDC 924472), $[Tb(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$ (CCDC 924473), $[Dy(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$ (CCDC 924474) [11], $[Ho(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$ (CCDC 924467) [12], $[Er(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$ (CCDC 925788) [13], $[Tm(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$ (CCDC 925790) [14], $[Yb(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$ (CCDC 924466) [15], and $[Lu(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$ (CCDC 925789) [16].

Continuing these studies, in this work we described the synthesis and structure of the new binuclar complex of spirocarbone and rare-earth metal using

Table 1. Main crystallographic experimental data for structure **I**

Parameter	Value
Empirical formula	$C_{22}H_{48}Ce_2N_{14}O_{26}$
FW	1204.98
Crystal system	Monoclinic
Space group	$P2_1/n$
Cell parameters:	
a, Å	14.2797(13)
b, Å	7.3922(6)
c, Å	21.003(2)
β, deg	102.103(10)
V, Å^3	2167.8(3)
Z	2
ρ_{calcd}, g/cm^3	1.85
$\mu(MoK_\alpha)$, mm^{-1}	2.18
$F(000)$	1204
Crystal size, mm	0.08 × 0.04 × 0.01
θ Range, deg	1.928–28.278
Ranges of reflection indices	$-18 \le h \le 19$, $-9 \le k \le 9$, $-28 \le l \le 28$
Number of measured/independent reflections (R_{int})	21163/5376 (0.1429)
Number of reflections with $I > 2\sigma(I)$	3661
Number of refined parameters	293
R factor ($I > 2\sigma(I)$)	$R_1 = 0.0921$, $wR_2 = 0.1930$
R factor (all reflections)	$R_1 = 0.1382$, $wR_2 = 0.2152$
Goodness-of-fit for F^2	1.107
$\Delta\rho_{max}$ and $\Delta\rho_{min}$, e Å^{-3}	3.055 and −3.379

cerium(III) nitrate as an example and discussed the change in the periodical properties in the series of the Sk complexes with lanthanide(III) (Ce–Lu, except Pm) nitrates.

EXPERIMENTAL

Cerium nitrate $Ce(NO_3)_3 \cdot 6H_2O$ (reagent grade), Sk obtained by a described procedure [17], and acetone (special purity grade) were used.

Synthesis of compound I. A weighed sample of cerium nitrate was dissolved in acetone, spirocarbone was introduced in a mole ratio of 1 : 2, and the mixture was magnetically stirred for 5–10 min. The obtained solution was filtered and left for several days to form crystals in a closed vessel. White crystals that formed were filtered off, washed with acetone, and dried in air. The yield was ~74% (based on the ligand).

For $[Ce(C_{11}H_{20}N_4O_2)(H_2O)_2(NO_3)_3]_2$

anal. calcd., %:	C, 21.93;	H, 4.02;	N, 16.27.
Found, %:	C, 21.95;	H, 4.01;	N, 16.25.

The elemental analysis of compound **I** was carried out on an EA-3000 analyzer (EuroVector, Italy). The IR spectra of spirocarbone and complex **I** were recorded in KBr pellets on a SPECTRUM ONE FT-IR spectrophotometer (PerkinElmer) in a region of 400–4000 cm^{-1}.

X-ray diffraction analysis. An experimental material for crystals of complex **I** was obtained on an Xcalibur 3 automated four-circle diffractometer (MoK_α radiation, λ = 0.71073 Å) at 293(2) K. The structure was solved by a direct method using the SHELX-97 program package [18]. The positions of hydrogen atoms were calculated geometrically and refined by the riding model with $U_{iso} = nU_{eq}$ of the bearing atom ($n = 1.5$ for water and methyl groups, $n = 1.2$ for other hydrogen atoms). The structure was refined by the full-matrix least-square method in the anisotropic approximation for non-hydrogen atoms for F^2.

The main experimental characteristics and unit cell parameters are presented in Table 1. Selected bond lengths and bond angles in the structure are given in Table 2.

The coordinates of atoms and other parameters of structure **I** were deposited with the Cambridge Crystallographic Data Centre (CCDC 985758; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

The following characteristic bands are observed in the IR spectra of complex **I** and spirocarbone (ν, cm^{-1}): for Sk, 3416 (H_2O); 3335, 3293, 3218 (HN); 3075, 2991, 2978 (CH_3, CH_2); 1653 (C=O, amide I); 1418 (C–N); for **I**, 3721 (H_2O); 3341 (NH); 2976, 2932, 2883 (CH_3, CH_2); 1631 (C=O, amide I); 1434 (C–N); 1505, 1252, 1035 [19–21] (NO_3).

The IR spectrum of complex **I** exhibits a shift by 22 cm^{-1} to the long-wavelength region of the absorption band corresponding to the stretching vibrations ν(C=O (amide **I**)), indicating that the Sk molecules are coordinated. The IR spectrum also exhibits a shift to the short-wavelength region of the bands caused by the vibrations ν_s(NH) and ν_{as}(NH), which is characteristic of the amino groups at the coordinated carbonyl [22]. The absorption bands ν_{s+as}(HOH) of water and a set of absorption bands of the coordinated Sk ligand are also observed. The free nitrate anion as a planar ion (point group D_3) has four main vibrational frequencies: frequencies of symmetrical stretching vibrations ν_s(NO) (1050–1060 cm^{-1}) and nonsym-

Table 2. Bond lengths (Å) and bond angles (deg) in structure **I***

Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
Ce(1)–O(1)	2.466(7)	O(1)–C(1)	1.246(13)	O(11)–N(7)	1.239(12)
Ce(1)–O(2)^{i}	2.366(7)	O(2)–C(7)	1.289(12)	N(1)–C(1)	1.341(13)
Ce(1)–O(3)	2.715(8)	O(3)–N(5)	1.261(12)	N(1)–C(2)	1.490(13)
Ce(1)–O(4)	2.663(8)	O(4)–N(5)	1.294(12)	N(2)–C(1)	1.366(14)
Ce(1)–O(6)	2.644(7)	O(5)–N(5)	1.223(12)	N(2)–C(4)	1.474(14)
Ce(1)–O(7)	2.617(8)	O(6)–N(6)	1.292(12)	N(3)–C(2)	1.469(12)
Ce(1)–O(9)	2.669(8)	O(7)–N(6)	1.280(11)	N(3)–C(7)	1.359(14)
Ce(1)–O(10)	2.669(8)	O(8)–N(6)	1.234(12)	N(4)–C(7)	1.324(14)
Ce(1)–O(12)	2.521(8)	O(9)–N(7)	1.270(11)	N(4)–C(8)	1.474(13)
Ce(1)–O(13)	2.546(7)	O(10)–N(7)	1.278(11)		
Angle	ω, deg	Angle	ω, deg	Angle	ω, deg
O(1)Ce(1)O(3)	69.7(3)	O(7)Ce(1)O(10)	66.9(2)	C(7)N(3)C(2)	126.2(9)
O(1)Ce(1)O(4)	75.4(3)	O(9)Ce(1)O(3)	129.0(2)	C(7)N(4)C(8)	124.2(9)
O(1)Ce(1)O(6)	135.2(3)	O(9)Ce(1)O(10)	47.8(2)	O(3)N(5)O(4)	115.0(9)
O(1)Ce(1)O(7)	129.3(3)	O(10)Ce(1)O(3)	168.7(2)	O(5)N(5)O(3)	123.9(10)
O(1)Ce(1)O(9)	154.6(3)	O(12)Ce(1)O(3)	122.3(2)	O(5)N(5)O(4)	121.0(10)
O(1)Ce(1)O(10)	117.3(2)	O(12)Ce(1)O(4)	148.7(3)	O(7)N(6)O(6)	115.9(9)
O(1)Ce(1)O(12)	73.6(3)	O(12)Ce(1)O(6)	144.8(3)	O(8)N(6)O(6)	122.2(9)
O(1)Ce(1)O(13)	63.7(3)	O(12)Ce(1)O(7)	135.8(3)	O(8)N(6)O(7)	121.9(9)
O(2)^{i}Ce(1)O(1)	96.9(3)	O(12)Ce(1)O(9)	81.2(3)	O(9)N(7)O(10)	116.3(9)
O(2)^{i}Ce(1)O(3)	68.0(3)	O(12)Ce(1)O(10)	68.9(3)	O(11)N(7)O(9)	122.6(10)
O(2)^{i}Ce(1)O(4)	113.7(3)	O(4)Ce(1)O(9)	129.4(3)	O(11)N(7)O(10)	121.0(9)
O(2)^{i}Ce(1)O(6)	81.4(3)	O(12)Ce(1)O(13)	86.7(3)	O(1)C(1)N(1)	122.1(10)
O(2)^{i}Ce(1)O(7)	127.1(2)	O(13)Ce(1)O(3)	113.3(3)	O(1)C(1)N(2)	120.1(10)
O(2)^{i}Ce(1)O(9)	78.8(3)	O(13)Ce(1)O(4)	76.1(3)	N(1)C(1)N(2)	117.8(10)
O(2)^{i}Ce(1)O(10)	117.7(3)	O(13)Ce(1)O(6)	121.7(3)	N(1)C(2)C(3)	106.1(8)
O(2)^{i}Ce(1)O(12)	74.2(3)	O(13)Ce(1)O(7)	76.4(3)	N(1)C(2)C(9)	111.3(8)
O(2)^{i}Ce(1)O(13)	156.3(3)	O(13)Ce(1)O(9)	112.5(3)	N(3)C(2)N(1)	108.2(8)
O(4)Ce(1)O(3)	47.2(2)	O(13)Ce(1)O(10)	65.8(3)	N(3)C(2)C(3)	107.7(8)
O(4)Ce(1)O(9)	129.4(3)	C(1)O(1)Ce(1)	136.6(7)	N(3)C(2)C(9)	108.8(8)
O(4)Ce(1)O(10)	124.0(3)	C(7)O(2)Ce(1)^{i}	169.3(7)	N(2)C(4)C(3)	106.8(8)
O(6)Ce(1)O(3)	68.3(2)	N(5)O(3)Ce(1)	98.0(6)	N(2)C(4)C(5)	108.8(9)
O(6)Ce(1)O(4)	65.0(3)	N(5)O(4)Ce(1)	99.6(6)	O(2)C(7)N(3)	119.2(10)
O(6)Ce(1)O(9)	69.4(3)	N(6)O(6)Ce(1)	96.4(5)	O(2)C(7)N(4)	121.4(10)
O(6)Ce(1)O(10)	102.2(2)	N(6)O(7)Ce(1)	98.1(6)	O(11)N(7)O(9)	122.6(10)
O(7)Ce(1)O(3)	101.9(2)	N(7)O(9)Ce(1)	98.0(6)	N(4)C(7)N(3)	119.4(10)
O(7)Ce(1)O(4)	65.4(3)	N(7)O(10)Ce(1)	97.8(6)	N(4)C(8)C(9)	106.2(9)
O(7)Ce(1)O(6)	48.9(2)	C(1)N(1)C(2)	126.3(9)	N(4)C(8)C(10)	110.8(8)
O(7)Ce(1)O(9)	68.7(3)	C(1)N(2)C(4)	128.0(9)	N(4)C(8)C(11)	108.9(9)

* Symmetry procedure: ^{i} $1-x, -y, 1-z$.

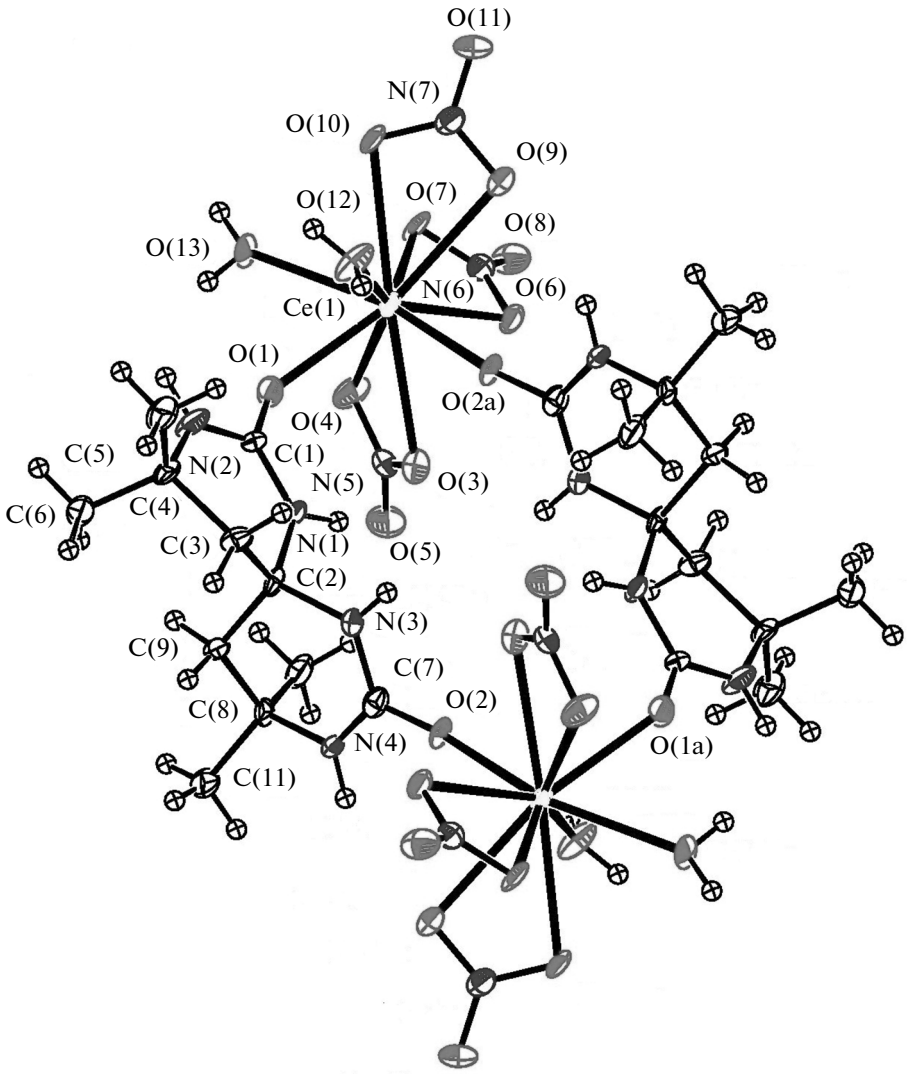


Fig. 1. Molecular structure of compound **I**.

metrical doubly degenerate stretching vibrations ν_e(NO) (1350–1400 cm^{-1}) and two frequencies of bending vibrations $\delta(NO_3)$ (810–840 and 710–730 cm^{-1}). Only three frequencies are usually active in the IR spectrum: ν_e(NO) and two $\delta(NO_3)$ [19]. For the coordination of the nitrate ion, its symmetry can decrease to C_s and C_{2v}. As a result, six intense lines appear in the IR spectrum (cm^{-1}): 970–1040 (full-symmetry vibration), 1550–1410 and 1290–1250 (stretching antisymmetrical vibration that appears as two intense bands), 830–800 (nonplanar vibration), and 780–700 and ~680 (planar bending vibration that appears as two bands) [20, 21]. The IR spectrum of compound **I** contains lines at 1505, 1252, and 1035 cm^{-1}. This indicates that the nitrate anions are coordinated according to the bidentate-chelating mode.

According to the X-ray diffraction data, compound **I** is a centrosymmetrical binuclear complex of the cerium(III) ion with two spirocarbone molecules bound by the symmetry procedure $(1 - x, -y, 1 - z)$, bidentate nitrate anions, and two water molecules (in the partial position) (Fig. 1). The crystals of complex **I** are isostructural to those of the lanthanum complex [8]. The coordination polyhedron of the metal atom is an irregular ten-vertex polyhedron. The Ce...Ce distance in the complex is 9.57 Å.

The six-membered heterocycles of spirocarbon are in the conformation, which is intermediate between a sofa and a twist bath. In the cycle containing the N(1) atom, the fragment N–C–N–C(Me_2) fragment is nearly planar (torsion angle –2°) and the C_{spiro} and C(H_2) atoms deviate to different sides from this plane by 0.21(2) and –0.39(2) Å, respectively. In the cycle containing the N(3) atom, the C(Me_2) and C(H_2) atoms deviate by –0.31(2) and 0.39(2) Å, respectively, from the plane of the C_{spiro}–N–C(–O)–N fragment (torsion angle –9.6°). This results in the shortened

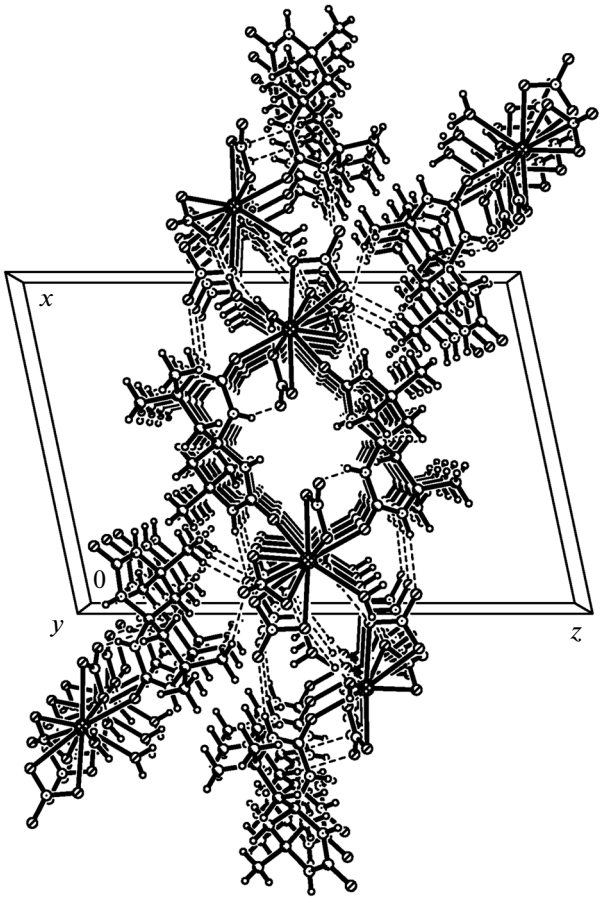


Fig. 2. Packing of structural units in crystal **I**.

intramolecular contacts H(3)...H(3b) 2.27 and H(6a)...H(9b) 2.28 Å (the doubled van der Waals radius of hydrogen is 2.32 Å [23]).

The weak hydrogen bond N(1)–H(1)...O(3)(H...O 2.48 Å, N–H...O 144°) is formed inside the complex.

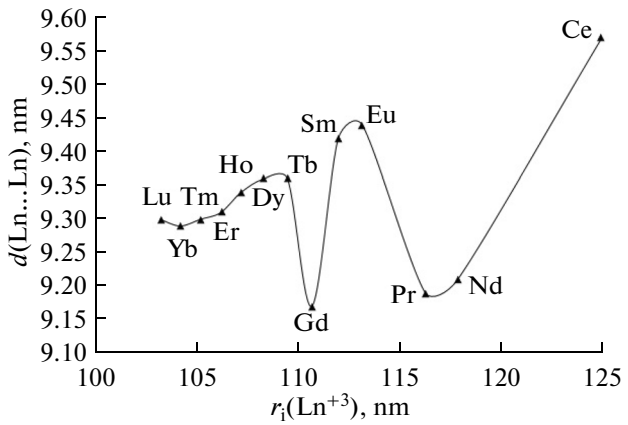


Fig. 3. Distance Ln…Ln in the lanthanide complexes vs. ion radius.

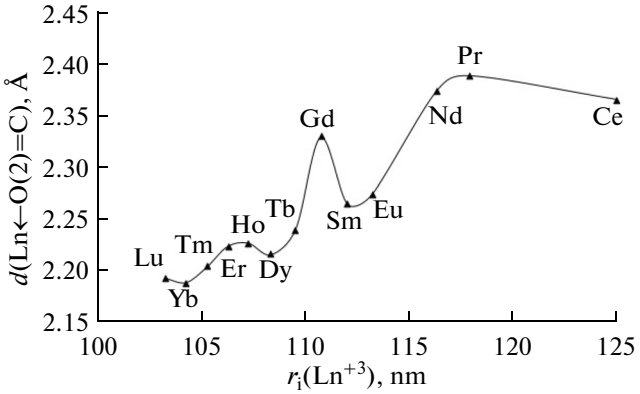


Fig. 4. Bond length Ln←O(2)=C in the lanthanide complexes vs. ion radius.

The complexes are bonded between each other into layers parallel to the plane (0 0 1) by intermolecular hydrogen bonds N(2)–H(2)...O(11)^{i} (^{i} $-x$, $-y$, $1-z$) (H...O 2.34 Å, N–H...O 162°), N(4)–H(4)...O(8)^{ii} (^{ii} $1-x$, $1-y$, $1-z$) (H...O 2.22 Å, N–H...O 170°), O(12)–H(12a)...O(10)^{i} (H...O 1.94 Å, N–H...O 174°), O(12)–H(12b)...O(4)^{iii} (^{iii} x, $-1+y$, z) (H...O 2.04 Å, O–H...O 166°), O(13)–H(13a)...O(7)^{iv} (^{iv} $-x$, $1-y$, $1-z$) (H...O 1.93 Å, O–H...O 172°) (Fig. 2).

We have previously [11] mentioned for the synthesized lanthanide (Sm–Dy) complexes the nonmonotonic character of changing the unit cell volume, the distance between the lanthanide atoms Ln···Ln, and bond lengths between the Ln atoms and carbonyl oxygen of spirocarbone at the coordination number of Ln equal to 9.

The generalized material [9–16] of X-ray structural data for all lanthanide complexes with spirocarbone (except for the Pm complex) is presented in Table 3.

Note that for all complexes of spirocarbone and lanthanides the nonmonotonic character of changing

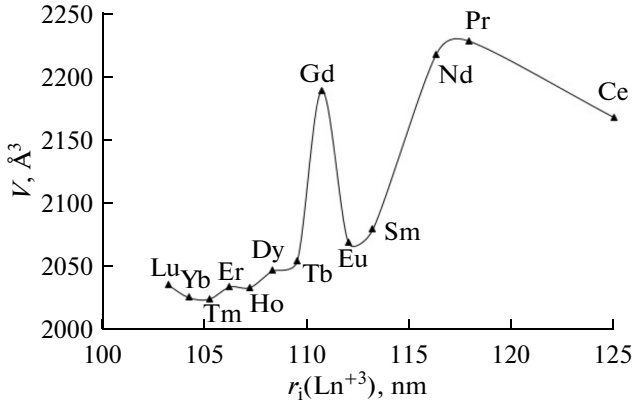


Fig. 5. Unit cell volume in the lanthanide complexes vs. ion radius.

Table 3. Selected parameters of the structures of the lanthanide and spirocarbone complexes

Atom Ln	$r_i(Ln^{+3})$, pm (Shannon [24])	d(Ln...Ln), nm	d(Ln←O(2)=C), Å	d(Ln–OH_2), Å	β angle, deg	V, Å^3	Literature
Ce	125.0	9.57	2.366	2.546	102.103	2167.80	
Pr	117.9	9.21	2.389	2.565	94.744	2228.32	[9]
Nd	116.3	9.19	2.374	2.545	94.800	2217.85	[10]
Sm	113.2	9.44	2.274	2.416	98.976	2079.20	[11]
Eu	112.0	9.42	2.264	2.419	98.822	2068.65	[11]
Gd	**110.7**	**9.17**	**2.329**	**2.479**	**94.697**	**2189.00**	**[11]**
Tb	109.5	9.36	2.240	2.384	98.583	2054.08	[11]
Dy	108.3	9.36	2.215	2.375	98.481	2046.50	[11]
Ho	107.2	9.34	2.226	2.360	98.363	2033.25	[12]
Er	106.2	9.31	2.223	2.344	98.144	2033.70	[13]
Tm	105.2	9.30	2.203	2.336	98.186	2024.02	[14]
Yb	104.2	9.29	2.188	2.325	98.195	2025.18	[15]
Lu	103.2	9.30	2.192	2.325	97.885	2034.99	[16]

the Ln...Ln distance, Ln bond length (carbonyl of Sk, water molecule), volume, and β angle of the unit cell is observed (Figs. 3–7).

In spite of the fact that in complex **I** the coordination number of the Ce atom is 10 and in other complexes the coordination number of Ln is 9, they can be divided into two isostructural subgroups:$[Ln(Sk)(H_2O)_3(NO_3)_2]_2(NO_3)_2$ (Pr(III), Nd(III), Gd(III) and $[LnSk(H_2O)(NO_3)_3]_2$ (Sm(III), Eu(III), Tb(III), Dy(III), Ho(III), Er(III), Tm(III), Yb(III), Lu(III)). The periodical properties are violated on the gadolinium atom in the coordination compounds of lanthanides with 4,4,10,10-tetramethyl-1,3,7,9-tetraazospiro[5.5]undecane-2,8-dione.

This violation of the monotonic character is a consequence of secondary periodicity named the tetrad effect [25, 26]. The reasons for this effect are considered to be the nonuniform rate of deepening of the 4*f* orbitals in the lanthanide series resulting in the "gadolinium break" and the spin-orbital interaction dividing the whole series of lanthanides into four segments: the additional stabilization by the crystalline field, which is maximum at the beginning and at the end of two subgroups of the lanthanide family.

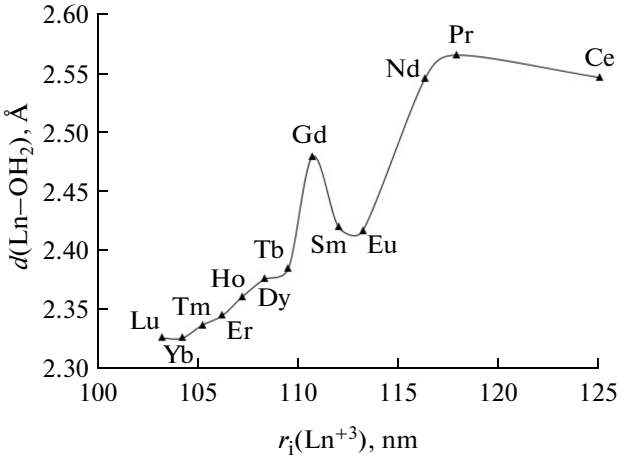


Fig. 6. Bond length Ln←OH_2 in the lanthanide complexes vs. ion radius.

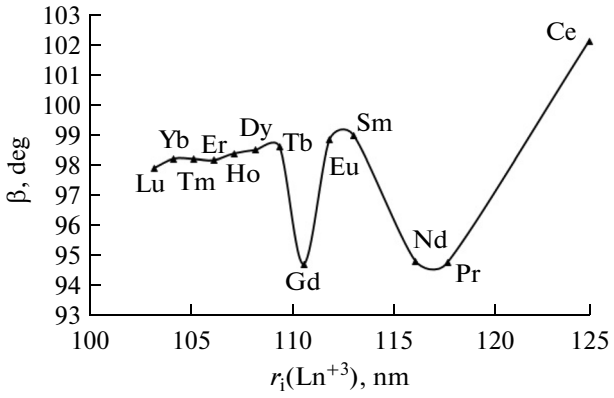


Fig. 7. Unit cell angle in the lanthanide complexes vs. ion radius.

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Translated by E. Yablonskaya