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Synthesis and Crystal Structure of the New Complex Bis(4,4,10,10-Tetramethyl-1,3,7,9-Tetraazospiro[5.5]undecane-2,8-Dione-O)diaquatris(nitrato-O,O')cerium(III)

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Abstract—A mononuclear biligand complex of cerium(III) nitrate with bicyclic bisurea (4,4,10,10-tetramethyl-1,3,7,9-tetraazospiro[5.5]undecane-2,8-dione or spirocarbone (Sk)), $Ce(C_{11}H_{20}N_4O_2)_2(H_2O)_2(NO_3)_3$ (**I**), is synthesized. Its structure is determined by X-ray diffraction analysis (CIF file CCDC 985759). The crystals are monoclinic: space group $P2_1/c$, a = 11.1935(5), b = 13.0139(5), c = 24.1857(10) Å, β = 101.146(4)°, V = 3456.7(3) Å^3, ρ_{calcd} = 1.619 g/cm^3, Z = 4. The structure is molecular. The cerium(III) cation is coordinated by two oxygen atoms of two molecules of the organic ligand, two $O(H_2O)$ atoms, and six O atoms of three bidentate nitrate anions. The coordination number of the cerium(III) atom is ten, and the coordination polyhedron is an irregular ten-vertex polyhedron. The crystal is a non-merohedral twin, whose components are turned by 180° along the x axis, and the relative weights of the components are 0.82 : 0.18.

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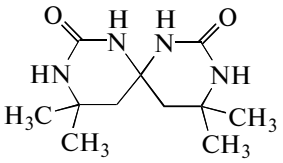
INTRODUCTION

Coordination compounds with ligands of the bicyclobisureas are poorly studied to the moment. One of these ligands is 4,4,10,10-tetramethyl-1,3,7,9-tetraazospiro[5.5]undecane-2,8-dione ($C_{11}H_{20}N_4O_2$), or spirocarbone ($C_{11}H_{20}N_4O_2$, Sk):



This bicyclospirobisurea is a precursor of urea and has a series of valuable biological properties: a low toxicity level LD_{50} = 3000 mg/(kg of weight of white mice) [1], membranotropicity [2], and the ability to penetrate through and be accumulated in the cytoplasm of leucosis cells of the mice and human L1210 and CEM-T4 lines, respectively [3]. Spirocarbone Sk also favors an increase in the amount of protein and a decrease in starchiness in oats [4]. The efficiency of spirocarbone as a stimulator of callus formation in Forsythia europaea [5] and root formation in Snowbelle was proved. The efficiency of using spirocarbone as a stimulator of growth and development in sheep breeding was shown [6]. Therefore, the synthesis and study of coordination compounds of this bicyclospirobisurea (hard Lewis base) will clarify more completely the chemical nature of the interaction of Sk with various metal atoms.

We have previously synthesized and characterized the binuclear complexes of spirocarbone with rare-earth metals in the oxidation state 3+ (Table 1) in which the metal to ligand ratio is 1 : 1. An analysis of the synthesis procedures of the earlier mentioned complexes showed that the specified molar ratio of the reactants, n(salt) > n(ligand), resulted in the formation of binuclear 16-membered chelates in which ligand Sk performed the bidentate-bridging function.

Continuing these studies, in this work we describe the synthesis and structure of the new mononuclear biligand complex of spirocarbone and cerium(III) nitrate, bis(4,4,10,10-tetramethyl-1,3,7,9-tetraazospiro[5.5]undecane-2,8-dione-O)diaqua-tris(nitrato-O,O')cerium(III) (**I**). The molar ratio of the reactants n(salt) < n(ligand) was specified in the synthesis.

EXPERIMENTAL

Cerium nitrate hexahydrare $Ce(NO_3)_3 \cdot 6H_2O$ (reagent grade), Sk obtained by known procedures [18, 19], and acetone (special purity grade) were used.

Synthesis of compound I. A weighed sample of cerium(III) nitrate was dissolved in acetone, spirocarbone was added in a molar ratio of 1 : 3, and the mixture was magnetically stirred for 10 min. The obtained solution was filtered, corked up, and left to stay for several days to form crystals. The formed white crystals were filtered off, washed with acetone, and dried in air. The yield was ~64% (based on the salt).

Table 1. Complexes with 4,4,10,10-tetramethyl-1,3,7,9-tetraazospiro[5.5]undecane-2,8-dione (Sk)

Complex with Sk	CCDC	Literature
$[Y(C_{11}H_{20}N_4O_2)(H_2O)_3(NO_3)_2]_2(NO_3)_2$	903389	[7]
$[La(C_{11}H_{20}N_4O_2)(H_2O)_2(NO_3)_3]_2$	903388	[8]
$[Pr(C_{11}H_{20}N_4O_2)(H_2O)_3(NO_3)_2]_2(NO_3)_2$	924475	[9]
$[Nd(C_{11}H_{20}N_4O_2)(H_2O)_3(NO_3)_2]_2(NO_3)_2$	876569	[10]
$[Sm(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$	924470	[11]
$[Eu(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$	924469	[11]
$[Gd(C_{11}H_{20}N_4O_2)(H_2O)_3(NO_3)_2]_2(NO_3)_2$	924472	[11]
$[Tb(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$	924473	[11]
$[Dy(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$	924474	[11]
$[Ho(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$	924467	[12]
$[Er(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$	925788	[13]
$[Tm(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$	925790	[14]
$[Yb(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$	924466	[15]
$[Lu(C_{11}H_{20}N_4O_2)(H_2O)(NO_3)_3]_2$	925789	[16]
$[Ce(C_{11}H_{20}N_4O_2)(H_2O)_2(NO_3)_3]_2$	985758	[17]

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

anal. calcd., %:	C, 31.35;	H, 5.26;	N, 18.28.
Found, %:	C, 31.37;	H, 5.25;	N, 18.30.

Analyses to C, H, and N in compound **I** were carried out on an EA-3000 elemental analyzer (EuroVector, Italy). The IR spectra of ligand Sk and synthesized compound **I** were recorded in KBr pellets on a Spectrum ONE FT-IR spectrophotometer (PerkinElmer) in a range of 400–4000 cm^{-1}.

X-ray diffraction analysis. An experimental material for a crystal of complex **I** was obtained on an Xcalibur 3 automated four-circle diffractometer (MoK_α radiation, $\lambda = 0.71073$ Å) at 293(2) K. The structure was solved by a direct method using the SHELX-97 program package [20]. 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 the H atoms of water and methyl groups, $n = 1.2$ for other hydrogen atoms). The structure was refined by full-matrix least squares in the anisotropic approximation for non-hydrogen atoms for F^2. The studied crystal of compound **I** was a non-merohedral twin, whose components were turned by 180° along the *a* axis, and the relative weights of the components were 0.82 : 0.18. The main experimental characteristics and unit cell parameters are presented in Table 2. Bond lengths and bond angles in structure **I** are given in Table 3.

Table 2. Selected crystallographic experimental data for structure **I**

Parameters	Value
Empirical formula	$C_{22}H_{44}N_{11}O_{15}Ce$
FW	842.80
Crystal system	Monoclinic
Space group	$P2_1/c$
Cell parameters:	
a, Å	11.1935(5)
b, Å	13.0139(5)
c, Å	24.1857(10)
β, deg	101.146(4)
V, Å^3	3456.7(3)
Z	4
ρ_{calcd}, g/cm^3	1.619
$\mu(MoK_\alpha)$, mm^{-1}	1.398
F(000)	1724
Crystal size, mm	0.074 × 0.182 × 0.289
θ angle range, deg	2.69–29.11
Ranges of reflection indices	$-14 \le h \le 15$, $-17 \le k \le 17$, $-32 \le l \le 33$
Number of measured/independent reflections (R_{int})	9039/9039 (0.0000)
Number of reflections with $I > 2\sigma(I)$	5483
Number of refined variables	457
R factor ($I > 2\sigma(I)$)	$R_1 = 0.0639$, $wR_2 = 0.1582$
R factor for all reflections	$R_1 = 0.1066$, $wR_2 = 0.1868$
Goodness-of-fit for F^2	0.928
$\Delta\rho_{max}/\Delta\rho_{min}$, *e* Å^{-3}	2.310/–2.804

The coordinates of atoms and other parameters for structure **I** were deposited with the Cambridge Crystallographic Data Centre (CIF file CCDC 985759; 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 compound **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** 3620 (H_2O); 3405, 3391, 3371, 3346 (NH); 2976, 2935, 2876 (CH_3, CH_2); 1657, 1652, 1628 (C=O, amide **I**); 1482, 1455, 1437 (C–N); 1536, 1502, 1250, 1034, 819, 735, 705, 648 (NO_3).

A comparison of the stretching vibrations ν(C=O, amide **I**) shows a shift of 25 cm^{-1} to the long-wavelength range due to the coordination of Sk molecules in structure **I**. The singlet is also split, because complex **I** has free C=O groups, and a shift to the short-wavelength range of ν_s(NH), ν_{as}(NH) and is observed, which is characteristic of the amino groups at the coordinated carbonyl [21]. The IR spectrum of complex **I** contains absorption bands ν_{s+as}(HOH) of water and a set of absorption bands of the coordinated Sk ligand. The free nitrate anion as a planar ion with the point group D_{3h} has four main vibration frequencies: symmetric stretching vibrationsν_s(NO) at 1050–1060 cm^{-1}, non-symmetric doubly degenerated stretching vibrations ν_e(NO) at 1350–1400 cm^{-1}, and two frequencies of bending vibrations δ(NO_3) at 810–840 and 710–730 cm^{-1}. Only three frequencies are usually active in the IR spectrum: one ν_e(NO) and two δ(NO_3) [22]. Upon coordination, the symmetry of the nitrate ion can decrease to C_s and C_{2v}. As a result, six intense lines appear in the IR spectrum [23]: the full-symmetry vibration at 970–1040 cm^{-1}, the stretching antisymmetric vibration split into two intense lines at 1550–1410 and 1290–1250 cm^{-1}, the nonplanar vibration in a range of 830–800 cm^{-1}, and the planar bending vibration appeared as two bands at 780–700 and ~680 cm^{-1} [24]. The IR spectrum of complex **I** exhibits lines at 1536, 1502, 1250, 1034, 819, 735, and 705 cm^{-1}. This proves that the nitrate anions are coordinated via the bidentate-chelating mode.

According to the X-ray diffraction data, compound **I** is a molecular complex in which the cerium(III) cation is coordinated by two oxygen atoms of two Sk molecules, two O atoms of the water molecules, and six O atoms of three bidentate nitrate anions. The coordination polyhedron of the Ce atom is an irregular ten-vertex polyhedron (Fig. 1).

Two Sk molecules have different conformations. The six-membered heterocycle containing the N(1) atom exists in a conformation intermediate between a half-chair and a twist boat with the flattened fragment N(1)–C(1)–N(2)–C(2) (the corresponding torsion angle is 6.7(14)°) and the deviation of the C(3) and

Table 3. Bond lengths (Å) and bond angles (deg) in structure **I**

Bond	*d*, Å	Bond	*d*, Å
Ce(1)–O(1)	2.490(7)	O(11)–N(11)	1.246(11)
Ce(1)–O(3)	2.452(6)	O(12)–N(11)	1.275(11)
Ce(1)–O(5)	2.612(6)	O(13)–N(11)	1.222(11)
Ce(1)–O(6)	2.646(7)	N(1)–C(1)	1.342(11)
Ce(1)–O(8)	2.671(6)	N(1)–C(4)	1.463(11)
Ce(1)–O(9)	2.653(7)	N(2)–C(1)	1.314(12)
Ce(1)–O(11)	2.578(7)	N(2)–C(2)	1.471(12)
Ce(1)–O(12)	2.664(7)	N(3)–C(4)	1.464(11)
Ce(1)–O(14)	2.502(6)	N(3)–C(7)	1.347(11)
Ce(1)–O(15)	2.578(7)	N(4)–C(7)	1.335(11)
O(1)–C(1)	1.275(11)	N(4)–C(8)	1.494(11)
O(2)–(7)	1.284(11)	N(5)–C(12)	1.327(11)
O(3)–C(12)	1.239(10)	N(5)–C(15)	1.486(11)
O(4)–C(18)	1.253(11)	N(6)–C(12)	1.361(12)
O(5)–N(9)	1.268(10)	N(6)–C(13)	1.467(12)
O(6)–N(9)	1.276(10)	N(7)–C(15)	1.457(11)
O(7)–N(9)	1.234(10)	N(7)–C(18)	1.346(12)
O(8)–N(10)	1.274(11)	N(8)–C(18)	1.322(12)
O(9)–N(10)	1.257(10)	N(8)–C(19)	1.471(12)
O(10)–N(10)	1.213(10)		
Angle	ω, deg	Angle	ω, deg
O(1)Ce(1)O(5)	95.9(2)	N(11)O(12)Ce(1)	94.8(6)
O(1)Ce(1)O(6)	134.0(2)	C(1)N(1)C(4)	125.9(8)
O(1)Ce(1)O(8)	68.1(2)	C(1)N(2)C(2)	121.9(8)
O(1)Ce(1)O(9)	87.6(2)	C(7)N(3)C(4)	123.7(8)
O(1)Ce(1)O(11)	80.7(2)	C(7)N(4)C(8)	126.2(8)
O(1)Ce(1)O(12)	71.6(2)	C(12)N(5)C(15)	126.0(8)
O(1)Ce(1)O(14)	139.3(2)	C(12)N(6)C(13)	125.5(8)
O(1)Ce(1)O(15)	144.1(2)	C(18)N(7)C(15)	122.5(8)
O(3)Ce(1)O(1)	71.5(2)	C(18)N(8)C(19)	128.6(8)
O(3)Ce(1)O(5)	65.89(19)	O(5)N(9)O(6)	118.3(8)
O(3)Ce(1)O(6)	108.0(2)	O(7)N(9)O(5)	120.5(8)
O(3)Ce(1)O(8)	117.8(2)	O(7)N(9)O(6)	121.2(8)
O(3)Ce(1)O(9)	158.8(2)	O(9)N(10)O(8)	116.3(8)
O(3)Ce(1)O(11)	70.3(2)	O(10)N(10)O(8)	122.8(8)
O(3)Ce(1)O(12)	111.5(2)	O(10)N(10)O(9)	120.9(9)
O(3)Ce(1)O(14)	69.3(2)	O(11)N(11)O(12)	116.7(8)
O(3)Ce(1)O(15)	133.6(2)	O(13)N(11)O(11)	121.1(10)
O(5)Ce(1)O(6)	49.1(2)	O(13)N(11)O(12)	122.2(10)
O(5)Ce(1)O(8)	73.4(2)	O(1)C(1)N(1)	120.5(9)
O(5)Ce(1)O(9)	114.3(2)	O(1)C(1)N(2)	121.0(8)
O(5)Ce(1)O(12)	167.1(2)	N(2)C(1)N(1)	118.4(8)
O(6)Ce(1)O(8)	72.9(2)	N(2)C(2)C(3)	105.7(8)

Table 3. (Contd.)

Angle	ω, deg	Angle	ω, deg
O(6)Ce(1)O(9)	83.6(2)	N(2)C(2)C(5)	106.7(8)
O(6)Ce(1)O(12)	138.7(2)	N(2)C(2)C(6)	110.1(8)
O(9)Ce(1)O(8)	47.6(2)	N(1)C(4)N(3)	107.9(7)
O(9)Ce(1)O(12)	63.1(3)	N(1)C(4)C(3)	108.7(7)
O(11)Ce(1)O(5)	134.6(2)	N(1)C(4)C(9)	106.9(7)
O(11)Ce(1)O(6)	144.2(2)	N(3)C(4)C(3)	112.5(7)
O(11)Ce(1)O(8)	141.0(2)	N(3)C(4)C(9)	106.8(7)
O(11)Ce(1)O(9)	110.7(3)	O(2)C(7)N(3)	119.9(8)
O(11)Ce(1)O(12)	48.3(2)	O(2)C(7)N(4)	119.2(8)
O(11)Ce(1)O(15)	85.5(2)	N(4)C(7)N(3)	120.9(9)
O(12)Ce(1)O(8)	98.2(2)	N(4)C(8)C(9)	105.7(7)
O(14)Ce(1)O(5)	78.0(2)	N(4)C(8)C(10)	110.4(8)
O(14)Ce(1)O(6)	70.4(2)	N(4)C(8)C(11)	105.0(7)
O(14)Ce(1)O(8)	142.7(2)	O(3)C(12)N(5)	120.5(9)
O(14)Ce(1)O(9)	131.8(2)	O(3)C(12)N(6)	120.2(8)
O(14)Ce(1)O(11)	76.2(2)	N(5)C(12)N(6)	119.2(8)
O(14)Ce(1)O(12)	113.4(2)	N(6)C(13)C(14)	107.1(8)
O(14)Ce(1)O(15)	66.7(2)	N(6)C(13)C(16)	108.0(8)
O(15)Ce(1)O(5)	116.9(2)	N(6)C(13)C(17)	109.7(9)
O(15)Ce(1)O(6)	70.0(2)	N(5)C(15)C(14)	107.3(7)
O(15)Ce(1)O(8)	106.0(2)	N(5)C(15)C(20)	113.1(8)
O(15)Ce(1)O(9)	66.5(2)	N(7)C(15)N(5)	107.2(7)
O(15)Ce(1)O(12)	74.5(2)	N(7)C(15)C(14)	109.4(8)
C(1)O(1)Ce(1)	134.2(6)	N(7)C(15)C(20)	107.0(7)
C(12)O(3)Ce(1)	131.5(6)	O(4)C(18)N(7)	120.8(9)
N(9)O(5)Ce(1)	97.0(5)	O(4)C(18)N(8)	120.8(9)
N(9)O(6)Ce(1)	95.1(5)	N(8)C(18)N(7)	118.4(9)
N(10)O(8)Ce(1)	97.4(5)	N(8)C(19)C(20)	108.2(7)
N(10)O(9)Ce(1)	98.7(6)	N(8)C(19)C(21)	107.8(8)
N(11)O(11)Ce(1)	99.8(6)	N(8)C(19)C(22)	110.2(8)

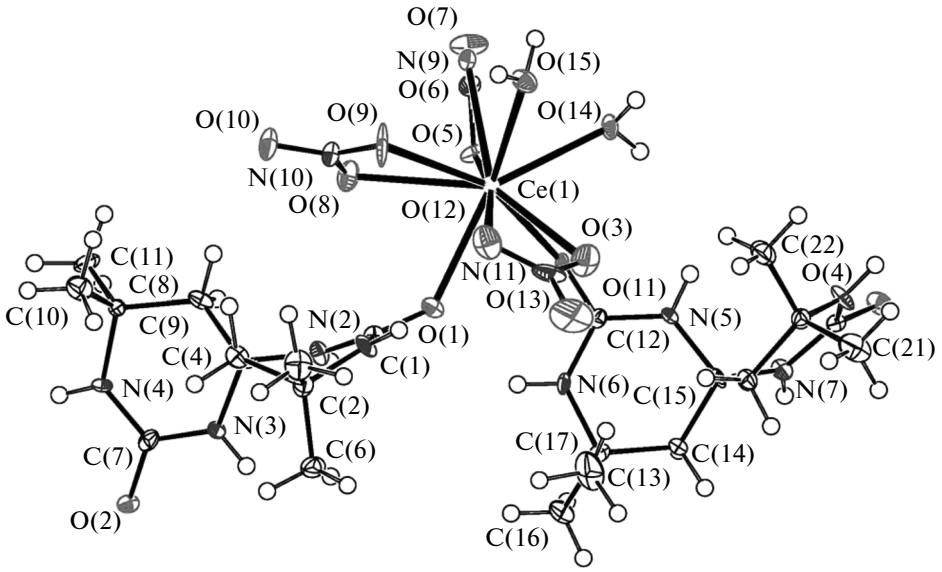


Fig. 1. Molecular structure of compound **I** according to the X-ray diffraction data.

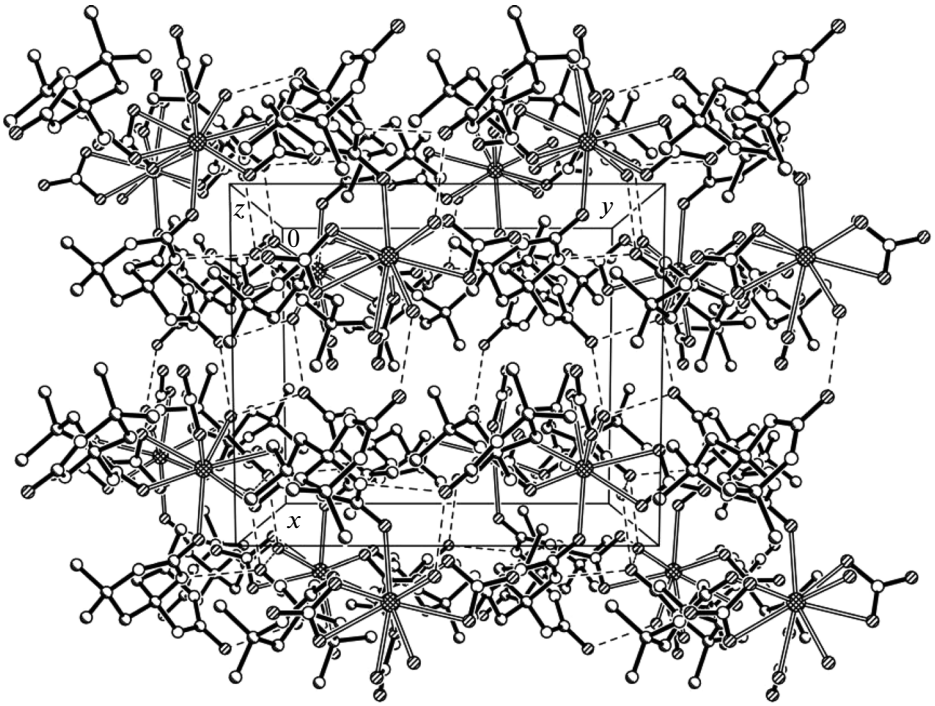


Fig. 2. Crystal structure of compound **I** according to the X-ray diffraction data.

Table 4. Hydrogen bonding geometry in structure **I**

D–H···A	Symmetry procedure	Distance, Å		Angle D–H···A, deg
		H···A	D···A	
O(14)–H(14*A*)···O(2)	$x, 1/2 - y, 1/2 + z$	1.92	2.738(9)	163
O(14)–H(14*B*)···O(2)	$-x, -1/2 + y, 1/2 - z$	1.92	2.744(10)	164
O(15)–H(15*A*)···O(4)	$-x, -y, 1 - z$	1.92	2.757(9)	166
O(15)–H(15*B*)···O(4)	$1 + x, y, z$	1.86	2.664(10)	156
N(2)–H(2)···O(12)		2.31	3.042(10)	144
N(3)–H(3)···O(3)	$-x, 1/2 + y, 1/2 - z$	2.49	3.214(10)	142
N(4)–H(4)···O(15)	$x, 1/2 - y, -1/2 + z$	2.18	3.033(10)	172
N(5)–H(5)···O(2)	$-x, -1/2 + y, 1/2 - z$	1.97	2.789(10)	160
N(6)–H(6)···O(1)		2.44	3.003(11)	124
N(7)–H(7)···O(9)	$-1 + x, y, z$	2.18	3.005(10)	161
N(8)–H(8)···O(6)	$-x, -y, 1 - z$	2.25	3.103(10)	174

C(4) atoms from the mean plane of this fragment by –0.89(2) and –0.58(2) Å, respectively. The cycles containing N(3), N(5), and N(7) atoms exist in the sofa conformation with the deviation of the C(9), C(14), and C(15) atoms from the planes of other atoms of the cycle by –0.59(1), –0.53(1), and –0.57(1) Å, respectively.

The crystal of complex **I** contains multiple intermolecular hydrogen bonds (Table 4) connecting the molecules of the complex between each other (Fig. 2).

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