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Crystal Structure of Mono- and Trinuclear Ni(II) Complexes with Bis-Azomethine Based on 2-Amino-3-Formylpyridine

Yu. P. Tupolova[a, *], **S. I. Levchenkov**[a, b], **L. D. Popov**[a], **S. V. Posokhova**[c], **and A. A. Aleksandrov**[d, †]
[a] *Southern Federal University, Rostov-on-Don, 344006 Russia*
[b] *Southern Scientific Center, Russian Academy of Sciences, Rostov-on-Don, Russia*
[c] *Azov Black Sea Engineering Institute, Don State Agrarian University, Zernograd, Rostov oblast, 347740 Russia*
[d] *Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences, Moscow, 119991 Russia*
**e-mail: yptupolova@sfedu.ru*

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Abstract—The structure of a new mononuclear nickel(II) complex with bis-azomethine $[Ni(H_2L)]^+ \cdot CH_3COO^- \cdot H_2O$, resulting from condensation of 1,3-diaminopropan-2-ol with 2-amino-3-formylpyridine (H_3L), was studied by X-ray diffraction (CIF files CCDC nos. 1562927, 1562928). Apart from the mononuclear complex, the reaction gave a minor amount of the trinuclear metal chelate $[Ni_3L'(H_2O)_2]^+ \cdot (CH_3COO)^- \cdot CH_3OH \cdot H_2O$, where L' is N_5O_2-donor ligand containing two 3-(((2-aminopyridin-2-yl)methylene)amino)-1-aminopropan-2-ol moieties linked by a 2-aminopyridine-3-methylene group.

Keywords: bis-azomethines, Ni(II) complexes, X-ray diffraction

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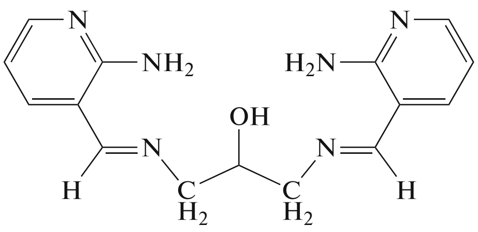
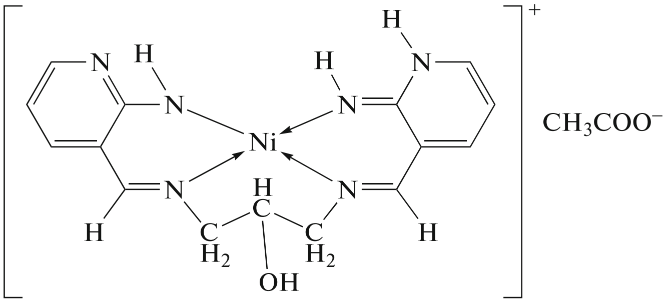
INTRODUCTION

1,3-Diaminopropan-2-ol bis-azomethines represent a vigorously studied ligand system [1]. These compounds are of interest for researchers, first of all, because they can be used to prepare binuclear complexes with unsymmetrical exchange moiety containing both mono- and bidentate exogenous bridging ligands. Deliberate variation of the nature of bridging groups can be used to study the mutual influence of the two exchange interaction channels connecting the paramagnetic centers of metal chelates; this is important for elucidation of magneto-structural correlations and finally for targeted synthesis of complexes with specified magnetic properties. An extensive experimental and theoretical study of the effects of electronic and geometric natures of the bridging groups was carried out and the key factors determining the strength and nature of exchange interactions in copper(II) complexes based on 1,3-diaminopropan-2-ol bis-azomethines were elucidated [2–12]. In addition, it was found that the structure and magnetic properties of metal chelates also depend on the aldehyde moiety of the ligand [13, 14]. Therefore, synthesis of new bis-azomethines and elucidation of the effect of their structural features on the structure and properties of complexes is a relevant task. In order to obtain a new ligand system based on 1,3-diaminopropan-2-ol as the carbonyl component, we chose 2-amino-3-formylpyridine, hydrazones and azomethines of which are scarcely reported in the literature [15–17].

This paper describes uncommon X-ray diffraction data for the mononuclear nickel complex (**I**), prepared by the reaction of nickel(II) acetate with bis-azomethine (H_3L), resulting from condensation of 2-amino-3-formylpyridine and 1,3-diaminopropan-2-ol.



(H_3L)



(**I**)

[†] Deceased.

EXPERIMENTAL

The starting compounds were commercially available chemicals. The solvents were purified and dried by standard procedures. IR spectra were recorded on a Varian Scimitar 1000 FT-IR instrument in the 400–4000 cm^{-1} range for samples as mineral oil mulls. The ^{1}H NMR spectra in DMSO-d$_6$ were run on a Varian Unity 300 spectrometer (300 MHz). Elemental analysis was carried out on a TCM 480 instrument (Carlo Erba Instruments). The mass spectrum of bis-azomethine H_3L was recorded on a Finnigan MAT INCOS 50 spectrometer (electron impact, 70 eV).

Synthesis of 1,3-bis(((2-aminopyridin-3-yl)methylene)amino)propan-2-ol (H_3L). A solution of 1,3-diaminopropan-2-ol (1 mmol) in methanol (15 mL) was added to a boiling solution of 2-amino-3-formylpyridine (2 mmol) in methanol (10 mL). The mixture was refluxed for 4 h and concentrated to a volume of 12 mL, and acetonitrile (15 mL) was added. The precipitated white solid was collected on a filter, washed with acetonitrile, and dried *in vacuo* at room temperature. The product was recrystallized from an acetonitrile–ethanol mixture (1 : 1). The yield was 0.42 g (70%). T_m = 145–146°C.

For $C_{15}H_{19}N_6O$			
Anal. calcd., %	C, 60.18	H, 6.40	N, 28.07
Found, %	C, 59.7	H, 6.61	N, 27.6

IR (ν, cm^{-1}): 3304 ν(OH), 3220 ν(NH), 1637, 1610, 1596 ν(C=N). ^{1}H NMR (DMSO-d$_6$; δ, ppm): 8.322 (s, 2H, CH=N), 7.99 (m, 2H, CH$_{\rm arom}$), 7.745 (br.s, 4H, NH$_2$), 7.626 (m, 2H, CH$_{\rm arom}$), 6.598 (m, 2H, CH$_{\rm arom}$), 4.986 (d, 1H, J = 5.1 Hz, OH), 3.97 (m, 1H, CH), 3.70 (m, 2H, CH$_2$), 3.58 (m, 2H, CH$_2$). MS: m/z 299, 298, 164, 135, 107.

Synthesis of I. A hot solution of nickel(II) acetate (1 mmol) in methanol (10 mL) was added to a hot solution of H_3L (1 mmol) in methanol (10 mL). The reaction mixture was refluxed for 1 h. The precipitate was collected on a filter and recrystallized from DMSO. The yield was 0.26 g (60%).

For $C_{17}H_{22}N_6NiO_4$			
Anal. calcd., %	C, 47.15	H, 5.12	N, 19.40
Found, %	C, 47.9	H, 5.01	N, 19.7

IR (ν, cm^{-1}): 3302 ν(OH), 3190 ν(NH), 1623, 1595 ν(C=N). ^{1}H NMR (DMSO-d$_6$, δ, ppm): 7.906 (m, 2H, CH$_{\rm arom}$), 7.755 (s, 2H, CH=N), 7.390 (d, 2H, J_1 = 7.8 Hz, J_2 = 1.8 Hz, CH$_{\rm arom}$), 6.153 (m, 2H, CH$_{\rm arom}$), 5.262 (d, 2H, J = 4.8 Hz, OH), 4.481 (s., 2H, NH), 4.08 (m, 1H, CH), 3.79 (m, 4 H, CH$_2$).

The X-ray diffraction study of I and II was carried out on a Bruker SMART APEX2 CCD diffractometer (MoK_α, λ = 0.71073 Å, graphite monochromator, ω-scan mode). The initial array of measured reflection intensities was treated using the SAINT and SADABS programs included in the APEX2 package [18, 19]. The structure was solved by direct methods and refined by the least squares method in the anisotropic approximation for non-hydrogen atoms on F^2_{hkl}. The hydrogen atoms were placed into the geometrically calculated positions. The structures were solved and refined using the SHELXTL program package [20] and analyzed using the PLATON program [21]. X-ray experimental details and crystallographic data are summarized in Table 1, selected interatomic distances and bond angles are listed in Table 2, and the geometric characteristics of hydrogen bonds are given in Table 3.

The atomic coordinates and thermal factors of structures **I** and **II** are deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 1562927, 1562928, respectively; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

The brown prismatic crystals of complex **I** suitable for X-ray diffraction were isolated directly from the reaction mixture. The molecular structure of complex **I** of the composition $[Ni(H_2L)]^+ \cdot CH_3COO^- \cdot H_2O$ is shown in Fig. 1. The monodeprotonated azomethine ligand is coordinated in the tetradentate fashion. In the coordinated bis-azomethine residue, both amino groups are deprotonated, but the pyridine group is protonated at N(2). The alcohol group is not involved in coordination. The O(1) atom and the methine C(14) atom are disordered over two crystallographic sites with equal occupancies.

The coordination polyhedron of nickel in complex **I** is a tetrahedrally distorted square; the angle between the N(4)Ni(1)N(6) and N(1)Ni(1)N(3) planes is 18.51°. The six-membered chelate ring Ni(1)N(1)C(1)C(5)C(6)N(3) has a asymmetric sofa conformation: the Ni(1) and N(3) atoms deviate from the plane through N(1), C(1), C(5), and C(6) atoms by 0.535 and 0.161 Å, respectively. The Ni(1)N(4)C(7)C(8)C(12)N(6) ring occurs in a similar conformation: the Ni(1) and N(4) atoms deviate from the C(7)C(8)C(12)N(6) plane by 0.351 and 0.123 Å, respectively. The third six-membered chelate ring Ni(1)N(3)C(13)C(14)C(15)N(4) is in the *twist*-conformation.

In the single crystal of complex **I**, one deprotonated amino group and the NH group of the protonated pyridine ring form hydrogen bonds, N(1)–H(1*A*)···O(1S) and N(2)–H(2*A*)···O(2S), with the acetate ion; weak N(6)–H(6*A*)···O(1S) hydrogen bond is also present (Table 3).

Table 1. Crystallographic data, X-ray experiment details, and structure refinement details for compounds **I** and **II**

Parameter	Value	
	I	**II**
Molecular formula	$C_{17}H_{22}N_6O_4Ni$	$C_{27}H_{37}N_{10}O_8Ni_3$
M	433.12	805.80
Crystal size, mm	0.33 × 0.12 × 0.10	0.19 × 0.17 × 0.06
Temperature of measurements, K	296(2)	296(2)
System	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1/c$
a, Å	9.6222(5)	14.919(2)
b, Å	9.9041(6)	16.871(2)
c, Å	10.5877(6)	14.2178(19)
α, deg	92.0910(8)	90
β, deg	97.7556(8)	114.971(2)
γ, deg	110.8292(7)	90
V, Å^3	930.53(9)	3244.1(8)
Z	2	4
ρ(calcd.), g/cm^3	1.546	1.650
μ, mm^{-1}	1.080	1.788
F(000)	452	1668
$2\theta_{max}$, deg	60.4	60.4
Ranges of reflection indices	$-13 < h < 13$, $-13 < k < 13$, $-14 < l < 14$	$-18 < h < 21$, $-21 < k < 23$, $-20 < l < 20$
Number of measured reflections	10726	29383
Number of unique reflections	5397	9584
Number of reflections with $I > 2\sigma(I)$	4596	4303
Number of refined parameters	297	448
GOOF (all reflections)	0.999	1.000
R_1 ($I > 2\sigma(I)$)	0.0316	0.0675
wR_2 (all reflections)	0.1072	0.2064
$\Delta\rho_{max}/\Delta\rho_{min}$, e Å^{-3}	0.361/–0.374	1.180/–0.812

The presence of solvate water molecule in the single crystal of **I** gives rise to three more hydrogen bonds: O(1)–H(1)···O(1–w)^{i}, O(1w)–H(1w1)···N(5)^{ii}, and O(1w)–H(2w1)···O(1S)^{ii} (crystallographic positions: ^{i} $-x$, $1 - y$, $1 - z$; ^{ii} $1 - x$, $2 - y$, $1 - z$). As a result, the single crystal of **I** is composed of layers of oppositely directed infinite zigzag-like chains of complex molecules, which are hydrogen-bonded through water molecules and acetate ions (Fig. 2).

According to the X-ray diffraction study, the sample of complex **I** isolated from the reaction mixture contains a minor (~5%) impurity of plate-like crystals of a different compound. It was shown that this impurity is the trinuclear complex $[Ni_3L'(H_2O)_2]^+ \cdot (CH_3COO)^- \cdot CH_3OH \cdot H_2O$ (**II**). Here L' is the N_5O_2-donor dodecadentate bridging ligand, which apparently results from partial hydrolysis of the bis-azomethine H_2L. It can be described as a geminal diamine composed of two 3-(((2-aminopyridin-2-yl)methylene)amino)-1-aminopropan-2-ol moieties cross-linked through the 2-aminopyridine groups by the 2-aminopyridine-3-methylene bridge. The bridging function of this ligand is performed by the alkoxide O(1) and O(2) atoms, which link Ni(1) to Ni(3) and Ni(2) to Ni(3), respectively, and by the amine N(10) atom, which links Ni(1) and Ni(2).

The solvate methanol molecule in **II** is disordered. It was impossible to locate the H atoms of one of the coordinated water molecules and the disordered sol-

Table 2. Selected interatomic distances and bond angles in the coordination polyhedra of the nickel atoms in structures **I** and **II**

Bond	*d*, Å	Bond	*d*, Å
I			
Ni(1)–N(6)	1.8439(14)	Ni(1)–N(4)	1.8839(14)
Ni(1)–N(1)	1.8669(14)	Ni(1)–N(3)	1.8925(13)
II			
Ni(1)–N(2)	1.837(5)	Ni(2)–N(6)	1.855(5)
Ni(1)–N(3)	1.864(5)	Ni(2)–N(7)	1.865(5)
Ni(1)–O(1)	1.871(4)	Ni(2)–O(2)	1.865(4)
Ni(1)–N(10)	1.914(5)	Ni(2)–N(10)	1.914(5)
Ni(3)–O(1)	2.045(4)	Ni(3)–O(2)	2.036(4)
Ni(3)–N(1)	2.063(6)	Ni(3)–N(5)	2.076(6)
Ni(3)–O(1*w*)	2.121(5)	Ni(3)–O(3*w*)	2.236(5)
Angle	ω, deg	Angle	ω, deg
I			
N(6)Ni(1)N(1)	88.44(6)	N(6)Ni(1)N(3)	166.94(6)
N(6)Ni(1)N(4)	92.23(6)	N(1)Ni(1)N(3)	91.77(6)
N(1)Ni(1)N(4)	166.39(6)	N(4)Ni(1)N(3)	90.63(6)
II			
N(2)Ni(1)N(3)	94.2(2)	N(6)Ni(2)N(7)	94.8(2)
N(2)Ni(1)O(2)	86.6(2)	N(6)Ni(2)O(2)	86.4(2)
N(3)Ni(1)O(1)	178.5(2)	N(7)Ni(2)O(2)	178.5(2)
N(2)Ni(1)N(10)	172.8(2)	N(6)Ni(2)N(10)	172.6(2)
N(3)Ni(1)N(10)	92.6(2)	N(7)Ni(2)N(10)	92.3(2)
O(1)Ni(1)N(10)	86.58(19)	O(2)Ni(1)N(10)	86.54(18)
N(2)Ni(1)Ni(2)	139.73(16)	N(6)Ni(2)Ni(1)	139.67(17)
N(3)Ni(1)Ni(2)	84.99(15)	N(7)Ni(2)Ni(1)	84.15(15)
O(1)Ni(1)Ni(2)	93.59(14)	O(2)Ni(2)Ni(1)	94.41(14)
N(10)Ni(1)Ni(2)	43.23(14)	N(10)Ni(2)Ni(1)	43.22(15)
O(2)Ni(3)O(1)	96.72(17)	N(1)Ni(3)N(5)	97.4(2)
O(2)Ni(3)N(1)	177.2(2)	O(1)Ni(3)N(5)	178.9(2)
O(1)Ni(3)N(1)	82.5(2)	O(2)Ni(3)N(5)	83.4(2)
O(2)Ni(3)O(1*w*)	88.2(2)	O(2)Ni(3)O(2*w*)	95.20(18)
O(1)Ni(3)O(1w)	89.0(2)	O(1)Ni(3)O(2*w*)	93.41(18)
N(1)Ni(3)O(1w)	89.1(2)	N(1)Ni(3)O(2*w*)	87.6(2)
N(5)Ni(3)O(1w)	90.0(3)	N(5)Ni(3)O(2*w*)	87.6(2)

vate methanol molecule. The molecular structure of complex **II** is shown in Fig. 3.

The coordination polyhedron of the Ni(1) and Ni(2) atoms in **II** is a slightly distorted planar square; the structural parameters of both atoms are rather close to each other (Table 2).

The six-membered chelate rings Ni(1)N(2)C(4)-C(5)C(9)N(3) and Ni(2)N(6)C(13)C(14)C(18)N(7) are somewhat distorted as a result of Ni atom deviations from the mean plane through other atoms by 0.242 and 0.182 Å, respectively. The six-membered chelate rings Ni(1)N(3)C(24)C(20)C(19)N(10) and Ni(2)N(7)C(24)C(20)C(19)N(10) are highly distorted via folding along the C(24)···N(10) line; the dihedral angles between the mean planes are 51.69° and 52.75°, respectively. The pyridine ring N(9)-

Table 3. Geometric parameters of hydrogen bonds in the single crystal of complexes **I** and **II***

D–H···A	Distances, Å			DHA angle, deg
	D–H	H···A	D···A	
		I		
N(1)–H(1*A*)···O(1S)	0.86	2.14	2.952(2)	158
N(2)–H(2*A*)···O(2S)	0.86	1.83	2.675(2)	168
N(6)–H(6*A*)···O(1S)	0.86	2.51	3.345(2)	165
O(1)–H(1)···O(1*w*)$^{\rm i}$	0.82	1.84	2.656(3)	175
O(1*w*)–H(1*w*1)···N(5)$^{\rm ii}$	0.81(2)	2.07(2)	2.851(3)	162(3)
O(1*w*)–H(2*w*1)···O(1S)$^{\rm ii}$	0.81(2)	1.99(2)	2.747(3)	158(3)
		II		
N(1)–H(1C)···O(2S)$^{\rm i}$	0.90	2.22	2.990(8)	143
O(1*w*)–H(1*w* 1)···O(3S)$^{\rm ii}$	0.82(8)	1.98(8)	2.784(11)	164(8)
O(1*w*)–H(2*w* 1)···O(3S)$^{\rm ii}$	0.82(4)	1.98(6)	2.746(8)	155(8)
N(5)–H(5*A*)···O(3*w*)$^{\rm ii}$	0.90	2.38	3.269(9)	172
N(5)–H(5*B*)···O(1S)$^{\rm i}$	0.90	2.31	3.168(8)	159

* Crystallographic positions: $^{\rm i}\,-x, 1-y, 1-z$; $^{\rm ii}\,1-x, 2-y, 1-z$ (**I**); $^{\rm i}\,-1+x, y, -1+z$; $^{\rm ii}\,-1+x, 1/2-y, -1/2+z$ (**II**).

C(19)C(20)C(21)C(22)C(23) is nearly perpendicular to the Ni(1)Ni(2)Ni(3) plane (the dihedral angle is 87.6°).

The conformation of the five-membered rings Ni(1)O(1)C(2)C(3)N(2) and Ni(2)O(2)C(11)C(12)N(6) can be described as an envelope, with the flap being formed by the methine C(2) and C(12) atoms, respectively.

The coordination polyhedron of Ni(3) is a distorted octahedron; the equatorial plane is formed by

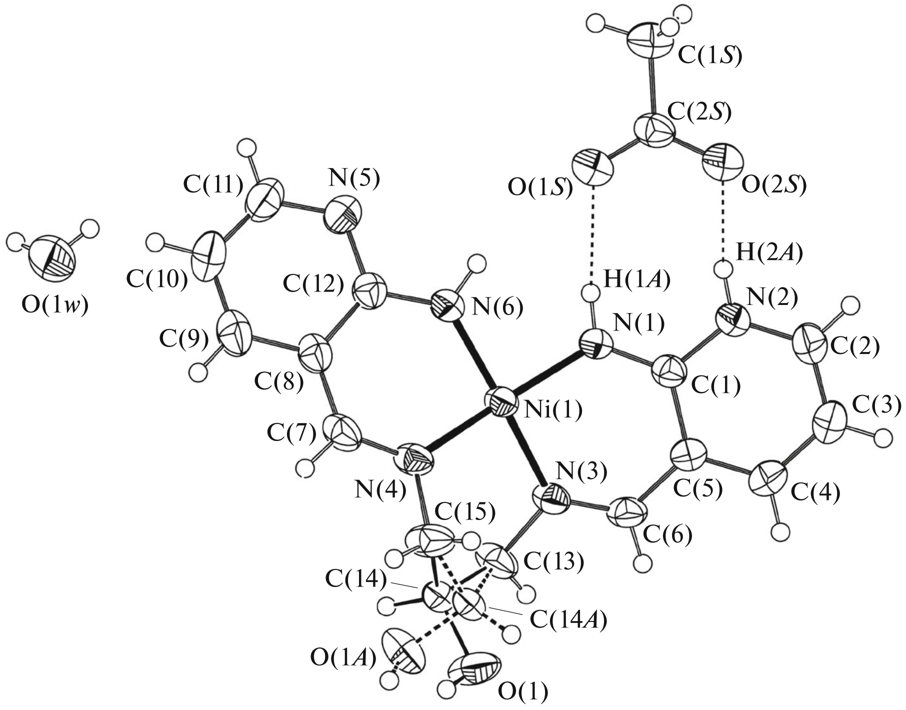


Fig. 1. Structure of the molecule of complex **I** with atoms represented as thermal ellipsoids drawn at 50% probability level.

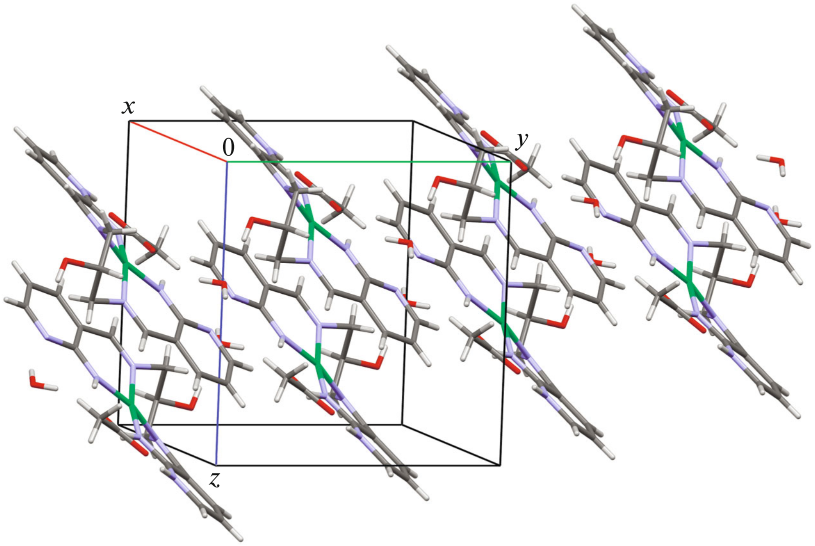


Fig. 2. Packing of molecules in the single crystal of complex **I** (view along the crystallographic x^* axis).

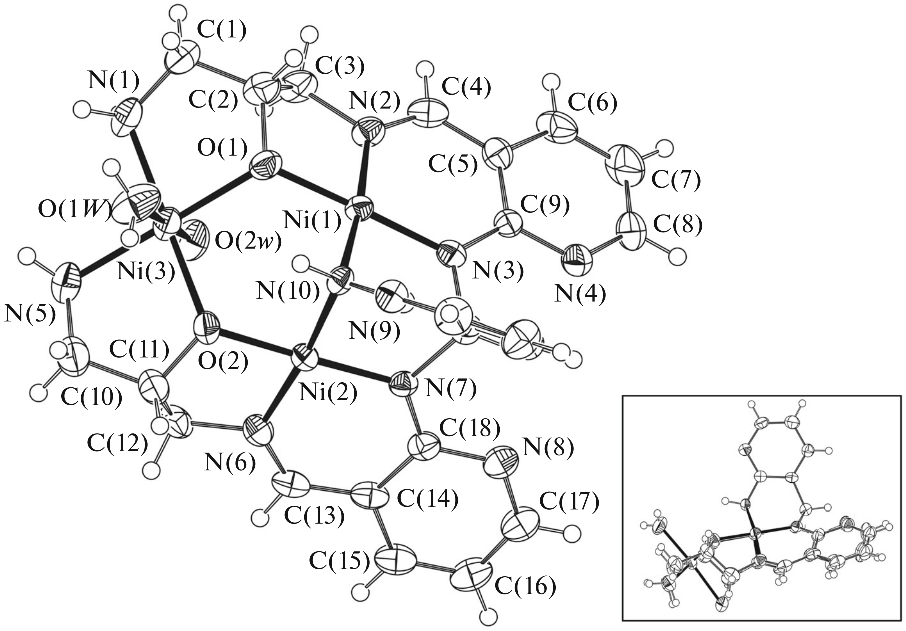


Fig. 3. Structure of the molecule of complex **II** with atoms represented as thermal ellipsoids at 50% probability level; the atoms of acetate ions and the solvate water molecules and methanol molecules are omitted. The inset shows the general view of the molecule along the Ni(1···Ni(2) line.

the bridging alkoxide O(1) and O(2) atoms and the N(1) and N(5) atoms, while the axial positions are occupied by O(1*w*) and O(2*w*) atoms of the coordinated water molecules. Both five-membered metallacycles Ni(3)N(1)C(1)C(2)O(1) and Ni(3)N(5)C(10)C(11)O(2) occur in the twist-conformation relative to the C–C bonds.

Generally, the molecular symmetry of complex **II** is quite similar to C_s (R = 0.1715). The Ni···Ni distances in complex **II** are markedly shorter than the sum of the van

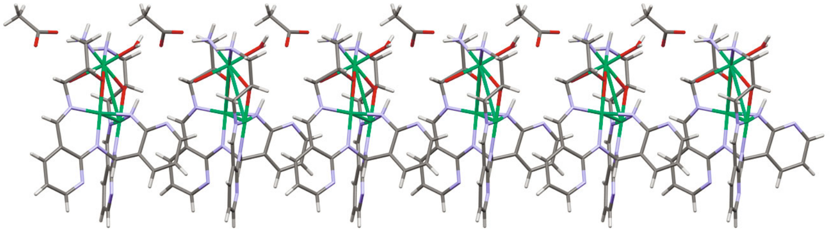


Fig. 4. Linear chains in the molecule of complex **II** in the single crystal (view along the crystallographic *y* axis).

der Waals radii, by ~30% for Ni(1)···Ni(2) (2.789(1) Å) and by ~15% for Ni(1)···Ni(3) and Ni(2)···Ni(3) (3.393(1) and 3.369(1) Å, respectively) [22–24].

In the single crystal of complex **II**, the molecules are connected by a branched system of hydrogen bonds (Table 3). By means of hydrogen bonds formed by the acetate ions and coordinated water molecules, the complex **II** molecules form linear chains extended along the crystallographic *z* axis (Fig. 4). The chains are connected to each other by hydrogen bonds involving solvate methanol molecules. Since the methanol molecules are disordered, the parameters of the corresponding bonds could not be determined.

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