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Nickel(II) and Copper(II) Complexes Based on *N*-(2-Carboxyethyl)alkanolamines: Influence of the Amino Alcohol Structure on the Coordination Sphere of the Metal Center

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Abstract—New nickel(II) complexes based on *N*-(3-hydroxypropyl)-β-alanine, *N*-(bis(hydroxymethyl)methyl)-β-alanine, and *N*-(tris(hydroxymethyl)methyl)-β-alanine are synthesized, and their structures are studied by X-ray diffraction analysis. The coordination spheres of the nickel and copper metal centers in the condensed phase are compared for a series of *N*-substituted β-alaninate ligands with the regularly changed dentate mode. In the case of the copper(II) complexes, an increase in the size of the alkanolamine chelate ring or the number of hydroxymethyl groups provides the formation of achiral coordination structures, whereas the structures of the nickel(II) complexes are independent of the size of the alkanolamine chelate ring or the number of hydroxymethyl groups, thus providing the formation of the complexes as racemic modifications.

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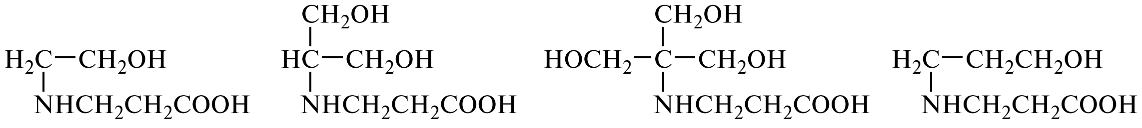
INTRODUCTION

An analysis of structures of coordination compounds, depending on the regular change in the chemical structure of the ligands, makes it possible to formulate conclusions that characterize the fundamental problems of their formation. The applied significance of their use, for example, as catalysts [1–3], biomimetic systems for studying the active centers of enzymes [4–6], and molecular electronics units [7–10] becomes more predictable. The complexes must contain ligands with a dentate mode of three and higher to accomplish these trends. Only in these situations the ligand can have a higher potential dentate mode, providing the necessary spatial arrangements of atoms, which finally results in the unique properties of these coordination compounds.

In spite of the relatively simple chemical structure, *N*-hydroxyalkyl derivatives of β-alanine, being tri- and more dentate ligands, provide the formation of transition metal polynuclear complexes [11–19]. To study the dependence of the structure of the copper(II) complexes on the regularly changed structure of the ligands (*N*-derivative of β-alanine), we considered a comparative series of ligands L^1–L^4 that monotonically increase the dentate mode due to the introduction of additional hydroxymethyl groups (scheme) [20].



$H_2C(NHCH_2CH_2COOH){-}CH_2OH$ (L^1)

$HC(CH_2OH)(NHCH_2CH_2COOH){-}CH_2OH$ (L^2)

$HOCH_2{-}C(CH_2OH)(NHCH_2CH_2COOH){-}CH_2OH$ (L^3)

$H_2C(NHCH_2CH_2COOH){-}CH_2CH_2OH$ (L^4)

Scheme.

In this work, we synthesized the new nickel(II) complexes using ligands L^2–L^4 and examined their compositions and structures with different ligands. A comparative analysis of the coordination sphere of the copper(II) and nickel(II) complexes based on ligands L^1–L^4 with the regularly changed dentate mode was performed.

EXPERIMENTAL

N-(Bis(hydroxymethyl)methyl)-β-alanine (L^2), *N*-(tris(hydroxymethyl)methyl)-β-alanine (L^3), and *N*-(3-hydroxypropyl)-β-alanine (L^4) were synthesized according to earlier developed procedures [13, 19, 20]. The synthesis of the nickel complex with ligand L^1 (**I**) has been described previously [21].

Synthesis of bis(*N*-(bis(hydoxymethyl)methyl)-β-alaninato)nickel(II) (II). A mixture of acid L^2 (1.0 g, 6 mmol), $(NiOH)_2CO_3$ (1.5 g, 7.1 mmol), and water (20 mL) was stirred at room temperature for 48 h. A precipitate was filtered off, and the filtrate was left at room temperature for the slow evaporation of water until light blue-green crystals precipitated.

For $C_{12}H_{24}N_2O_8Ni \cdot H_2O$

anal. calcd., %: C, 36.00; H, 6.50; N, 7.00; Ni, 14.50.

Found, %: C, 36.16; H, 6.89; N, 6.40; Ni, 14.95.

IR (ν, cm^{-1}): 3379 ν(NH), 3230 ν(OH), 1598 ν_{as}(C=O), 1422 ν_s(C=O).

Synthesis of [(*N*-(tris(hydroxymethyl)methyl)-β-alaninato)(aqua)(pyridine)nickel(II)] chloride (III). A mixture of chlorohydrate of acid L^3 (0.8 g, 3.5 mmol), $(NiOH)_2CO_3$ (0.81 g, 3.85 mmol), and water (20 mL) was stirred at room temperature for 48 h. A precipitate was separated, and the filtrate was evaporated to dryness.

For $C_7H_{14}NO_5ClNi \cdot 0.5H_2O$

anal. calcd., %: C, 28.52; H, 5.09; N, 4.75; Cl, 12.05; Ni, 19.69.

Found, %: C, 28.27; H, 5.25; N, 4.83; Cl, 12.55; Ni, 19.94.

IR (ν, cm^{-1}): 3255 ν(OH), 1563 ν_{as}(C=O), 1457 ν_s(C=O).

MS (ESI, H_2O–CH_3CN), *m/z*:
304^+ ($C_7H_{14}NNiO_5 + 3H_2O$),
537^+ ($[C_7H_{14}NNiO_5][C_7H_{13}NNiO_5] + 2H_2O$),
997^+ ($[C_7H_{14}NNiO_5][C_7H_{13}NNiO_5]_3$),
498^- ($[C_7H_{13}NNiO_5][C_7H_{12}NNiO_5]$),
997^- ($[C_7H_{13}NNiO_5]_3[C_7H_{12}NNiO_5]$).

The crystallization of a dry residue from an aqueous solution of pyridine gave blue crystals of complex **II**.

Synthesis of bis(*N*-(3-hydroxypropyl)-β-alaninato)nickel(II) (IV). $NiCl_2 \cdot 6H_2O$ (0.24 g, 1 mmol) in water (5 mL) was added with stirring to a mixture of acid L^4 (0.29 g, 2 mmol) and NaOH (0.12 g, 3 mmol) in water (5 mL). The resulting solution was left for slow crystallization at room temperature until light blue-green crystals precipitated.

For $C_{12}H_{24}N_2O_6Ni \cdot 3.5H_2O$

anal. calcd., %: C, 34.86; H, 7.50; N, 6.78; Ni, 14.04.

Found, %: C, 34.94; H, 7.13; N, 6.66; Ni, 14.40.

IR (ν, cm^{-1}): 3409 ν(NH), 3261 ν(OH), 1589 ν_{as}(C=O), 1430 ν_s(C=O).

Analyses to C, H, and N were carried out on an automated analyzer (PerkinElmer), and Ni was analyzed by atomic emission spectroscopy on an Optima 4300 DV spectrometer. IR reflectance spectra were recorded on a Spectrum-One spectrometer (PerkinElmer). The mass spectrometric characterization of the complexes was performed on a Shimadzu LCMS-2010 liquid chromatograph combined with a mass spectrometer in a water–acetonitrile (9 : 1) mixture using electrospray ionization.

X-ray diffraction analysis. The experimental data for compounds **II–IV** were obtained on an Xcalibur 3 automated diffractometer (CCD detector, MoK_α radiation, graphite monochromator, $T = 295(2)$ K). An absorption correction was applied analytically using the polyhedral crystal model [22]. The structure was solved and refined using the SHELX program package [23]. All non-hydrogen atoms were determined by a direct method and refined in the anisotropic approximation. Hydrogen atoms were placed in the geometrically calculated positions and included in the riding model with dependent thermal parameters.

The crystallographic data and experimental and refinement characteristics for structures **II–IV** are listed in Table 1. Selected bond lengths of the coordination node are given in Table 3. The atomic coordinates and thermal parameters for structures **II–IV** were deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 873439–873441; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk/data_request/cif).

RESULTS AND DISCUSSION

Complex $Ni(L^2)_2$ was synthesized by the reaction of basic nickel(II) carbonate and compound L^2 and crystallized as a 1 : 1 crystal hydrate (**II**). Its two potentially tetradentate ligands form the octahedral environment of the central ion and perform the tridentate function, closing two chelate cycles at each Ni–N bond: six-membered β-alaninate and five-membered ethanolamine (Fig. 1). The crystal represents a racemic mixture of two enantiomers due to the meridian conformation of the monodeprotonated ligands, providing the possibility of chiral center formation. A crystal of compound **II** contains a branched network of hydrogen bonds including different variants of intra- and intermolecular interactions between the molecules of the complex and between the complex and water of crystallization.

According to the elemental analysis data, the reaction of chlorohydrate of compound L^3 with basic nickel carbonate affords complex $Ni(L^3)Cl$. Attempts to obtain a single crystal of this compound for X-ray diffraction analysis was unsuccessful, most likely, because of the formation of a polymer coordination structure. Indeed, the mass spectrometric study of an aqueous solution showed the presence of dimeric and tetrameric associates. The crystallization of complex $Ni(L^3)Cl$ from an aqueous solution of pyridine gave blue crystals of complex **III**, and its crystal and molecular structures were studied by X-ray diffraction analysis. Complex **III** (Fig. 2) is mononuclear, the metal center is in the octahedral coordination environment, and the equatorial plane is formed of two hydroxyl

Table 1. Crystallographic data and experimental and refinement parameters for structures **II**–**IV**

Parameter	Value		
	II	**III**	**IV**
M	401.06	383.47	459.14
Crystal system	Triclinic	Monoclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1/n$	$C2/c$
Z	2	4	4
a, Å	8.4378(18)	13.8096(9)	16.6421(16)
b, Å	9.410(2)	8.3699(4)	10.1227(13)
c, Å	11.822(2)	13.9477(9)	12.4447(12)
α, deg	75.732(18)	90.00	90.00
β, deg	85.199(17)	107.407(6)	101.078(8)
γ, deg	65.32(2)	90.00	90.00
V, Å^3	826.4(3)	1538.31(16)	2057.4(4)
ρ_{calcd}, g/cm^3	1.612	1.656	1.482
μ, mm^{-1}	1.224	1.465	1.003
F(000)	424	800	984
Crystal sizes, mm	0.15 × 0.09 × 0.03	0.15 × 0.09 × 0.02	0.33 × 0.25 × 0.17
θ Range, deg	2.66–26.37	2.87–26.39	2.75–28.28
Ranges of reflection indices	$-10 \le h \le 9$, $-11 \le k \le 11$, $-13 \le l \le 14$	$-17 \le h \le 16$, $-10 \le k \le 7$, $-16 \le l \le 17$	$-22 \le h \le 15$, $-12 \le k \le 13$, $-15 \le l \le 16$
Measured reflections	9076	7426	7649
Independent reflections	3324	3131	2538
Reflections with $I \ge 2\sigma(I)$	2189	1938	1940
Number of refined parameters	241	223	155
R_1 ($I > 2\sigma(I)$)	0.0351	0.0339	0.0258
wR_2 ($I > 2\sigma(I)$)	0.0536	0.0597	0.0553
R_1 (all reflections)	0.0726	0.0615	0.0366
wR_2 (all reflections)	0.0584	0.0616	0.0566
Goodness-of-fit (all reflections)	1.008	1.002	1.004
Residual electron density (max/min), e/Å^3	0.404/–0.570	0.686/–0.372	0.380/–0.327

groups, the carboxylate group of ligand L^3, and water molecule. The nitrogen atoms of the amino group of the ligand and pyridine molecules lie on the axial axis. Thus, the potentially pentadentate ligand performs the tetradentate function. The complex exists in the cationic form, and the out-of-sphere chlorine ion serves as a counterion.

The use of ligand L^4 for the reaction with basic nickel(II) carbonate gave no complex. The synthesis of complex **IV** and the preparation of its single crystal for X-ray diffraction analysis became possible when using the exchange interaction of nickel(II) chloride and ligand L^4 as a sodium salt. As a result, complex **IV** was synthesized with the composition $Ni(L^4)_2 \cdot 6H_2O$. A molecule of the crystalline complex occupies the partial position on axis 2. The monodeprotonated ligands perform the tridentate-chelating function closing two six-membered chelate cycles at each Ni–N bond: β-alaninate and 3-aminopropanol (Fig. 3). In complex **IV**, the ligands have the meridian conformation to form the octahedral chiral environment of the metal center; that is, *N*-(3-hydroxypropyl)-β-alanine also interacts with the nickel(II) ion being in a conformation approaching that of the free ligand [13]. Crystal **IV** includes the branched network of hydrogen bonds involving six solvate water molecules (Table 3). Four solvate water molecules are arranged at the corners of the rectangle (O···O 2.901(2) and 2.903(2) Å) similarly to the earlier observed ordering in the case of the nickel complex of *N*-(2-pyridyl)methyl-3-aminopropionic acid [18].

A comparison of the coordination sphere of synthesized nickel(II) complexes **II**–**IV** shows (Fig. 4) that an increase in the dentate mode by the introduction of hydroxymethyl groups does not change the structures of the chelates, unlike the regularity of formation of the polynuclear complexes in similar copper complexes with ligands L^1–L^4 (**V**–**VIII**) [20]. No certain structure of the dimeric nickel(II) complex with ligand L^3 was determined by X-ray diffraction analysis because of a weaker complex formation with nickel(II) ions compared to the complexation ability of copper(II) cations. However, additional data of mass spectrometry show the possibility of formation of these associates in an aqueous solution. As in the case of the copper complexes, the coordination number of the nickel metal center remains unchanged (octahedral environment) with a monotonic increase in the number of hydroxymethyl groups. The same two chelate cycles are formed in each complex: six-membered β-alaninate and five-membered ethanolamine. This initially equalizes the complexes in strength. Ligand L^2 behaves rather strangely in the case of both copper complex **VI** and nickel complex **II**. The hydroxymethyl group can participate in coordination similarly to that occurring in the case of ligand L^3. However, L^2 exhibits the same reactivity as L^1. It is most likely that the entropy factor can also be used to explain the observed change in complex formation similarly to a series of the copper(II) complexes [20].

Table 2. Selected bond lengths (Å) and bond angles (deg) in the coordination nodes of structures **II–IV**

Bond	*d*, Å	Angle	ω, deg
		II	
Ni(1)–O(1)	2.0443(17)	O(1)Ni(1)O(3)	174.31(7)
Ni(1)–O(3)	2.0714(18)	O(5)Ni(1)O(7)	171.34(6)
Ni(1)–O(5)	2.0432(18)	N(1)Ni(1)N(2)	170.67(9)
Ni(1)–O(7)	2.1125(19)	O(5)Ni(1)O(1)	91.40(7)
Ni(1)–N(1)	2.067(2)	O(5)Ni(1)O(3)	91.48(7)
Ni(1)–N(2)	2.087(2)	O(5)Ni(1)N(1)	95.80(9)
		III	
Ni(1)–O(1)	2.0425(17)	O(1)Ni(1)O(4)	172.18(7)
Ni(1)–O(3)	2.060(2)	O(6)Ni(1)O(3)	174.08(10)
Ni(1)–O(4)	2.087(2)	N(1)Ni(1)N(2)	169.25(9)
Ni(1)–O(6)	2.063(2)	O(3)Ni(1)O(1)	93.95(8)
Ni(1)–N(1)	2.052(2)	O(1)Ni(1)O(6)	89.07(8)
Ni(1)–N(2)	2.074(2)	O(1)Ni(1)N(1)	90.24(7)
		IV	
Ni(1)–O(1)	2.0621(9)	O(1)Ni(1)N(1)	93.13(4)
Ni(1)–O(3)	2.0842(10)	O(1)Ni(1)O(3)	175.02(4)
Ni(1)–N(1)	2.0805(11)	O(3)Ni(1)N(1)	91.85(4)

A comparison of the conformations of the ligands in a series of nickel (**I–IV**) and copper (**V–VIII**) complexes shows that the nickel complexes exist in racemic modifications. Three copper(II) complexes (except for compound **VI**) demonstrate the achiral structure. Thus, in a series of *N*-hydroxyalkyl β-alaninate ligands, the nickel cation is prochiral, unlike the copper cation. Complex **VIII** with ligand L^4 is a dimer of the alkoxy derivative, and complex **VII** with ligand L^3 is also an alkoxy derivative but a cubane-like tetramer. Nickel hydroxide ($pK_b = 4.6$) is a stronger base than copper ($pK_b = 6.4$) and, hence, on going from the nickel(II) to copper(II) complexes with the same ligands, the oxo and hydroxo derivatives are formed.

A comparison of the characteristic bond lengths in the series of complexes **II–IV** shows (Table 4) that the

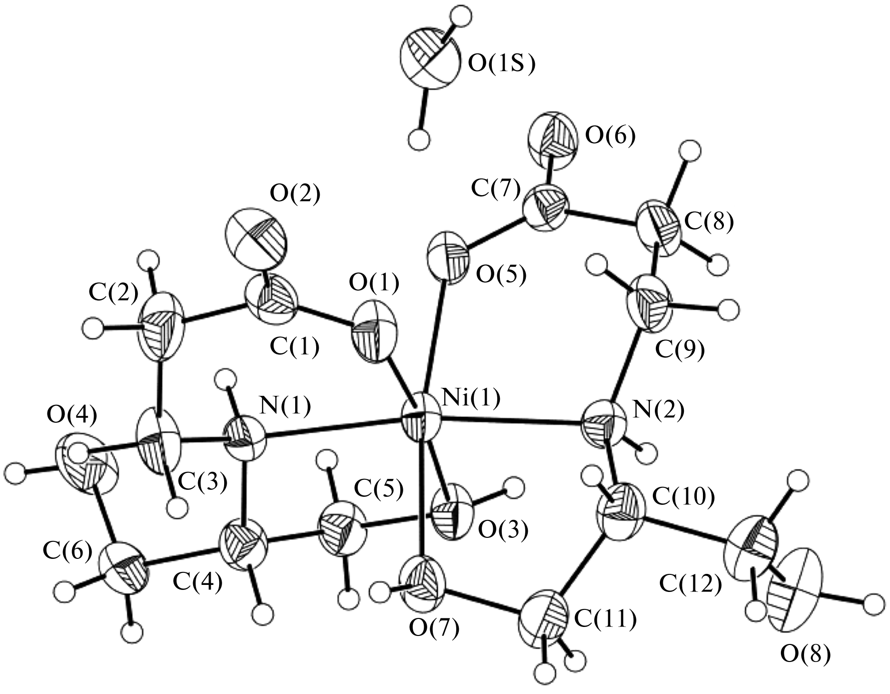


Fig. 1. Molecular structure of complex **II** in thermal ellipsoids of 50% probability.

Table 3. Geometric parameters of hydrogen bonds in complexes **II–IV** (for H···A < *r*(A) + 2 Å and angle >110°)

D–H···A	Distances, Å			Angle DHA, deg	Coordinates of atom A
	D–H	H···A	D···A		
II					
O(1S)–H(1S)···O(2)	0.88	1.91	2.768(5)	168	$-x, -y, -z + 2$
O(1S)–H(2S)···O(1)	0.96	2.21	3.153(5)	167	x, y, z
N(1)–H(1)···O(4)	0.87(3)	2.17(3)	2.999(3)	158(2)	$-x + 1, -y, -z + 1$
N(2)–H(2)···O(8)	0.88(2)	2.36(2)	2.820(3)	112(2)	x, y, z
O(7)–H(7)···O(2)	0.89(3)	1.73(3)	2.618(3)	171(2)	$-x + 1, -y, -z + 2$
O(4)–H(4)···O(5)	0.87(3)	1.86(3)	2.667(3)	153(2)	$-x + 1, -y, -z + 1$
O(3)–H(3)···O(6)	0.90(3)	1.69(3)	2.583(3)	168(2)	$-x, -y + 1, -z + 1$
O(8)–H(5)···O(1S)	0.90(4)	1.85(4)	2.728(3)	163(2)	$x, y + 1, z$
III					
O(6)–H(6*C*)···Cl(1)	0.80(3)	2.34(3)	3.127(3)	169(3)	x, y, z
O(4)–H(4)···O(2)	0.80(3)	1.84(3)	2.640(3)	178(3)	$x - 0.5, -y + 0.5, z - 0.5$
N(2)–H(2)···Cl(1)	0.93(3)	2.39(3)	3.284(3)	160(2)	x, y, z
O(6)–H(6*A*)···O(2)	0.82(3)	1.99(3)	2.802(3)	179(3)	$-x + 1, -y + 1, -z + 2$
O(5)–H(5)···Cl(1)	0.75(3)	2.40(4)	3.143(3)	178(4)	$x, y - 1, z$
IV					
O(6)–H(6S*B*)···O(4)	0.83(2)	2.09(2)	2.904(2)	169(2)	x, y, z
O(6)–H(6S*A*)···O(5)	0.73(2)	2.18(2)	2.901(2)	177(2)	$-x + 1.5, -y - 0.5, -z + 2$
O(5)–H(5S*A*)···O(6)	0.86(2)	2.05(2)	2.903(2)	172(2)	x, y, z
O(5)–H(5S*B*)···O(2)	0.74(2)	2.06(2)	2.802(2)	178(2)	$-x + 1, y, -z + 1.5$
O(4)–H(4S*B*)···O(5)	0.86(3)	2.04(3)	2.904(2)	175(2)	$-x + 1.5, y + 0.5, -z + 1.5$
O(4)–H(4S*A*)···O(2)	0.77(2)	2.18(2)	2.939(2)	171(2)	$-x + 1.5, y + 0.5, -z + 1.5$
O(3)–H(3)···O(2)	0.80(2)	1.90(2)	2.696(2)	173(2)	$x, -y, z + 0.5$
N(1)–H(1)···O(6)	0.86(2)	2.59(2)	3.353(2)	149(2)	x, y, z

Ni–OOC, Ni–N, and Ni–OH bonds in the N-substituted molecules of β-alanine and amino acids are shorter than those in the unsubstituted ligands, indicating the strengthening of the complexes. An increase in the dentate mode in the series of ligands $L^1 \rightarrow L^3$ also decreases the Ni–OOC and Ni–OH bond lengths, indicating the enhancement of the stability of the complexes, but elongates the Ni–N bond, which is likely caused by an increase in steric hindrances with a successive increase in the number of hydroxymethyl groups. An increase in the size of the five-membered ethanolamine chelate cycle to six, that is, going from ligand L^1 to L^4, changes neither the composition, nor structure of the nickel(II) complex, unlike the copper metal center [13]. This strengthens the Ni–OH bond only and weakens the coordination bonds of the β-alaninate chelate cycle.

Thus, the regularities of the influence of the dentate mode of the *N*-hydroxyalkyl β-alaninate ligands and the length of the chelate cycles on the composition and structure of the copper(II) and nickel(II) complexes were established. In the case of the copper metal center, an increase in the size of the *N*-(2-carboxyethyl)alkanolamine chelate cycle or an increase

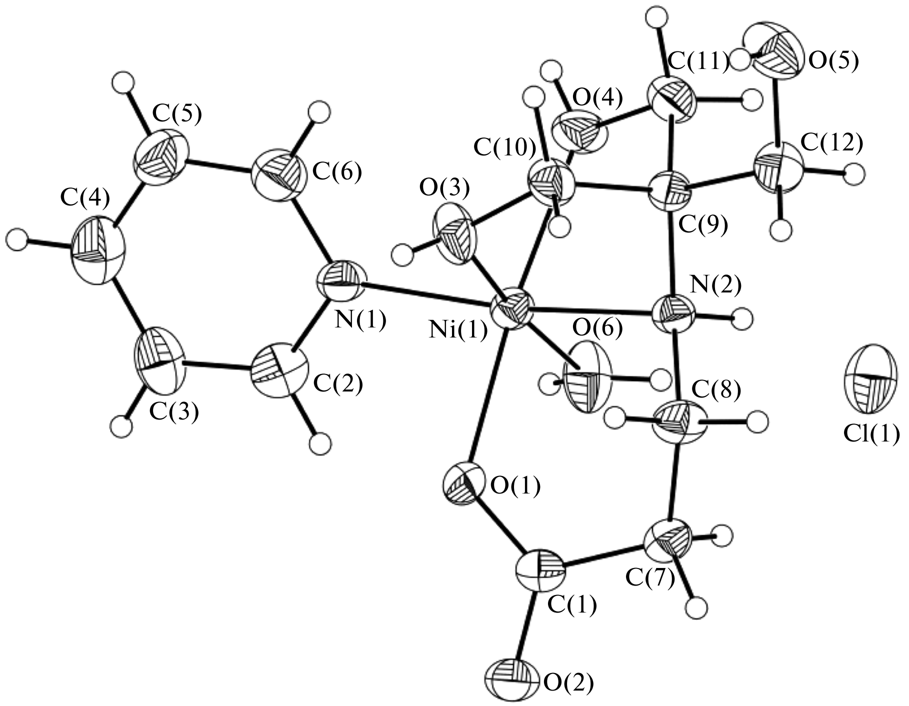


Fig. 2. Molecular structure of complex **III** in thermal ellipsoids of 50% probability.

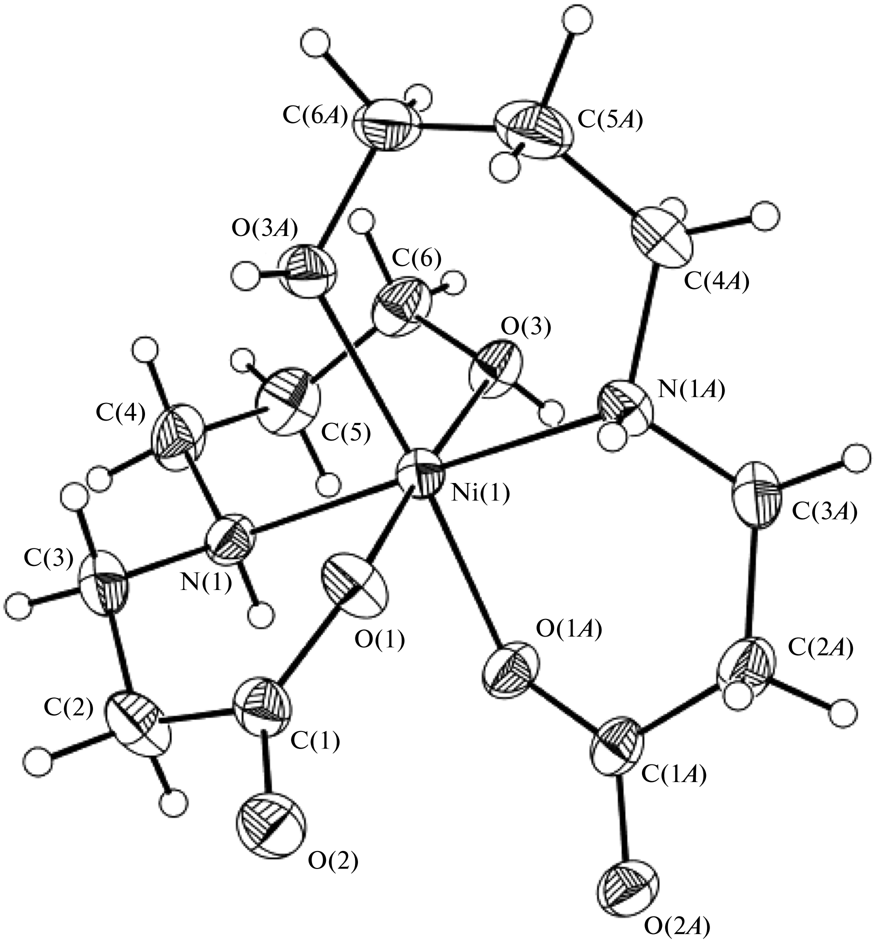
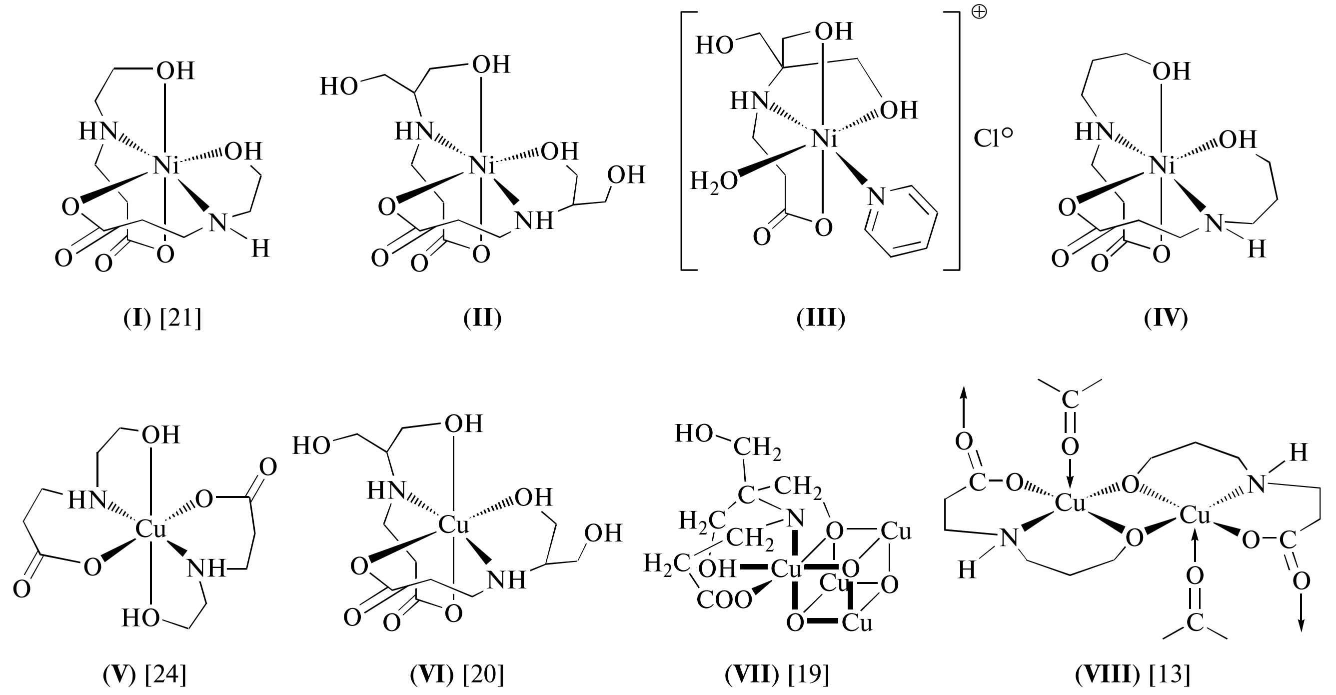


Fig. 3. Molecular structure of complex **IV** in thermal ellipsoids of 50% probability. Atoms of water of crystallization are omitted.



(**I**) [21] (**II**) (**III**) (**IV**)

(**V**) [24] (**VI**) [20] (**VII**) [19] (**VIII**) [13]

Fig. 4. Structures of the nickel(II) and copper(II) complexes formed by ligands L^1–L^4.

Table 4. Lengths of selected coordination bonds (Å) in the nickel(II) complexes with various 2-aminoethanol and β-alanine derivatives

Formula of ligand	Ni–OH	Ni–N	Ni–OOC	Literature
$H_2C{-}CH_2OH$ NH_2	2.141	2.102		[25]
$H_2C{-}CH_2{-}CH_2OH$ NH_2	2.096	2.085		[26]
$H_2NCH_2CH_2COOH$		2.075	2.085	[27]
$H_2C{-}CH_2OH$ $NHCH_2CH_2COOH$	2.097–2.134	2.037–2.045	2.026–2.069	[21]
CH_2OH $HC{-}CH_2OH$ $NHCH_2CH_2COOH$	2.071–2.112	2.067–2.087	2.043–2.044	The present article
CH_2OH $HOCH_2{-}C{-}CH_2OH$ $NHCH_2CH_2COOH$	2.087	2.074	2.042	The present article
$H_2C{-}CH_2{-}CH_2OH$ $NHCH_2CH_2COOH$	2.084	2.080	2.062	The present article
$H_2C{-}CH_2OH$ NCH_2CH_2COOH CH_2CH_2COOH	2.053–2.104	2.031	2.039	[28]

in the number of hydroxymethyl groups results in the formation of the achiral monochelates stabilized due to the formation of oligomers or polymers. The structure of the formed nickel monochelates is independent of the size of the hydroxyl-containing cycle and of an increase in the number of hydroxymethyl groups and provides the formation of the complexes in the racemic modification. In other words, the nickel(II) ion, unlike the copper(II) ion, is prochiral in this series of ligands.

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