COORDINATION COMPOUNDS =

Cobalt(II), Nickel(II), and Copper(II) Complexes with Schiff Bases, Derivatives of 1-Amino-8-hydroxynaphthalene-2,4-disulfonic Acid

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Abstract—Five new cobalt(II), nickel(II), and copper(II) complexes with Schiff bases have been synthesized. The Schiff bases have been prepared by the condensation of monopotassium 1-amino-8-hydroxynaphthalene-2,4-disulfonate with benzoin (L^1) or 2-hydroxy-1-naphthaldehyde (L^2) . The compounds have been identified and studied by elemental analysis, X-ray diffraction, thermogravimetry, measurements of magnetic susceptibility and electrical conductivity, and IR, ESR, and diffuse reflectance spectroscopy. The dimeric (with oxo bridges) structure of the Co(II) complex with L^1 has been additionally confirmed by the EXAFS method.

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The present study is intended as a continuation of our works devoted to the development of different methods of the synthesis and investigation of the composition and structure of coordination compounds of bivalent cobalt, nickel, and copper with the products of condensation of aminonaphthalene derivatives and aromatic carbonyl-containing compounds [1–6].

The products of condensation of aminonaphthalene(di)sulfonic acids, which are polydentate ligands, play a special role among diverse chelating Schiff bases. When studying the complex formation of Cu(II) 1-amino-8-hydroxynaphthalene-2,4-disulfonic with acid (1,8,2,4-ahKsHs'n) and benzoin (L¹) derivatives, we synthesized a square-planar complex in which the sulfo groups are not involved in coordination [5]. It was of interest to reveal whether the sulfo groups will be bonded when Cu(II) is replaced by Co(II) or Ni(II) (for which complexes with the bonds between the complexforming ion and the oxygen atom of the sulfo group were prepared [6]) and to elucidate how the change in the ligand system of L^2 (1,8,2,4-ahKsHs'n and 2-hydroxy-1-naphthaldehyde derivative) will affect the composition and structure of the Co(II), Ni(II), and Cu(II) complexes.

EXPERIMENTAL

The Schiff base-condensation product of 1-amino-8-hydroxynaphthalene-2,4-disulfonic acid (1,8,2,4ahKsHs'n) and benzoin [5]—was used as a ligand (L^1) for the synthesis of Ni(II) and Co(II) complexes (I and II, respectively; Table 1).

Synthesis of complexes I and II. Solutions of L^1 (1.65 g, 0.003 mol) in an ethanol-methanol (50 mL +70 mL) mixture and divalent nickel or cobalt (for I and **II**, respectively) chloride hexahydrate (0.71 g, 0.003 mol) in ethanol (20 mL) were mixed and refluxed in a water bath for 2 h. The resulting precipitates of complexes I and II were separated by filtration, washed with ethanol, and dried at room temperature above anhydrous calcium chloride to a constant weight.

Synthesis of Schiff base L². Solutions of 1,8,2,4ahKsHs'n (7.14 g, 0.02 mol) in ethanol (60 mL) and 2-hydroxy-1-naphthaldehyde (3.44 g, 0.02 mol) in ethanol (50 mL) were preliminarily mixed, and anhydrous sodium acetate (6 g, 0.07 mol) was added. The resulting solution was refluxed in a water bath for 1 h. The resulting dark brown precipitate of L² was subjected to the same procedures as those for the synthesis of complexes I and II. The melting point of the product is 185°C.

For C₂₁H₁₄KNO₈S₂ anal. calcd. (%): C, 49.32; H, 2.74; N, 2.74; S, 12.52; K, 7.63.

Found (%): C, 49.61; H, 2.60; N, 2.56; S, 12.68; K, 7.31.

Synthesis of complexes III–IV. Reaction mixtures consisting of L^2 (1.53 g, 0.003 mol) in ethanol (50 mL) and crystal hydrate of nickel(II), cobalt(II), or copper(II) chloride (0.003 mol; 0.71, 0.71, or 0.51 g, respectively) in ethanol (15 mL) were refluxed in a water bath for 1 h. Precipitates of complexes III–V formed upon cooling down were subjected to the same

	Calar	Empirical formula	Content (found/calculated), %					Molar electrical
Compound	Color		C	Н	Ν	S	M ²⁺	S cm ² mol ^{-1}
I*	Light green	$C_{48}H_{42}N_2O_{20}S_4Ni_2$	$\frac{47.63}{47.52}$	$\frac{3.51}{3.47}$	$\frac{2.50}{2.31}$	$\frac{10.28}{10.56}$	$\frac{9.60}{9.74}$	13.2
II	Lilac	$C_{48}H_{34}N_2O_{16}S_4Co_2$	$\frac{50.23}{50.52}$	$\frac{3.03}{2.98}$	$\frac{2.23}{2.46}$	$\frac{10.94}{11.23}$	$\frac{10.18}{10.35}$	11.6
III**	Dark green	$C_{42}H_{26}K_2N_2O_{16}S_4Ni$	$\frac{46.02}{46.71}$	$\frac{2.50}{2.41}$	$\frac{2.38}{2.59}$	$\frac{11.21}{11.86}$	$\frac{4.97}{5.47}$	140.2
IV*,**	Claret	$C_{42}H_{30}K_2N_2O_{18}S_4Co$	$\frac{44.96}{45.20}$	$\frac{2.46}{2.69}$	$\frac{2.34}{2.51}$	$\frac{11.19}{11.48}$	$\frac{5.52}{5.29}$	138.6
V **	Dark blue	$C_{42}H_{28}K_2N_2O_{16}S_4Cu$	$\frac{46.28}{46.43}$	$\frac{2.69}{2.58}$	$\frac{2.41}{2.58}$	$\frac{11.43}{11.79}$	$\frac{5.60}{5.85}$	146.4

Table 1. Characteristics of complexes I-V

Notes: * H₂O (found/calculated), %: 5.76/5.94 (I); 3.09/3.23 (IV).

** K (found/calculated), %: 6.80/7.23 (III); 6.73/7.00 (IV); 7.01/7.19 (V).

procedures as those for the syntheses of L^2 and complexes I and II.

The results of elemental analysis and some physicochemical characteristics of complexes **I–V** are given in Table 1.

The content of cobalt, nickel, and copper was determined by spectral X-ray fluorescence analysis on a SPARK-1 spectrophotometer with copper radiation in the 12 kV-10 mA mode with a counting rate of 400 pulse/s. The potassium content was determined by flame photometry.

Thermogravimetric analysis was carried out on a Q derivatograph (Paulik-Paulik-Erdey) in air. Samples were heated at a rate of 10 degree/min in the temperature range from 20 to 500°C. The reference was α -Al₂O₃.

X-ray powder diffraction patterns of the compounds were obtained on a DRON-3 diffractometer (Cu K_{α} radiation, Ni filter). The interplanar spacings were determined using tables [7].

Diffuse reflectance spectra were measured on a Perkin-Elmer UV VIS VIR Lambda 9 spectrophotometer in a range of 3000–30000 cm⁻¹ using MgO as the standard.

IR spectra were recorded as KBr pellets in the 200–4000 cm⁻¹ range on a Perkin-Elmer IR spectrophotometer.

Magnetic susceptibility was determined by the Gouy method at 293 K. The standard for calibration was $Hg[Co(NCS)_4]$.

The ohmic resistance of millimolar dimethylformamide solutions of complexes I-V for the calculation of the molar electrical conductivity was measured using an E7-8 digital resistance meter within 0–10 m Ω in an Arrhenius vessel. The ESR spectrum was recorded on an SE/X-2542 radiospectrometer (Radiopan) with a working frequency of 9.4 GHz. The magnetic field scan was calibrated using a nuclear magnetometer. The stable radical of diphenylpicrylhydrazyl was used as the reference for the determination of the *g* factor. Studies were carried out in a DMF solution. The concentration of the complex was 1×10^{-2} mol/L. Since a broad band caused by the Jahn–Teller effect was observed at room temperature in the spectrum of the studied complex [8], the measurements were carried out at 77 K.

The Co *K*-edge X-ray absorption was obtained in the transmission mode on an EXAFS spectrometer at the Siberian Center of Synchrotron Radiation (Novosibirsk, Russia). The energy of an electron beam used as a source of X-ray synchrotron radiation was 2 GeV at an average current of 80 mA. A double-crystal Si(111) monochromator was used to monochromatize X-ray radiation. The intensities of the incident and passed X-ray radiation were detected using ionization chambers filled with argon.

The sample was prepared for recording the EXAFS spectrum and exact parameters of the structure of the nearest environment of the cobalt atom were determined as described in [5]. The standards for fitting of structural parameters were theoretical phases and scattering amplitudes calculated for the bis(μ_2 -1,2-bis(2-hydroxybenzamido)benzene)bis(*N*-methylimidazole-*N*')dicobalt(II) complex [9], whose X-ray diffraction data were borrowed from the Cambridge Structural Database (CSD) [10].

RESULTS AND DISCUSSION

Ligand L^1 was used for the synthesis of compounds I and II [5]. Complexes III–V were synthesized from

III		I	V	V		
<i>d</i> , Å	<i>I</i> // ₀ , %	<i>d</i> , Å	<i>I</i> / <i>I</i> ₀ , %	<i>d</i> , Å	<i>I</i> / <i>I</i> ₀ , %	
1.404	13	1.62	30	1.41	13	
1.62	16	1.99	48	1.63	18	
1.99	47	2.28	39	2.03	21	
2.80	100	3.15	100	2.27	11	
3.26	13	3.56	22	2.76	100	
7.20	15	5.39	52	5.46	11	

Table 2. X-ray diffraction patterns for complexes III–V

L², which is the condensation product of 1,8,2,4-ahK-sHs'n and 2-hydroxy-1-naphthaldehyde. In the composition of preparatively isolated complexes I-V, the M^{2+} : L molar ratio was 1 : 1 in complexes I and II and 1 : 2 in complexes III-V, although this ratio was 1 : 1 in all the starting reaction mixtures.

Compounds I and II are X-ray amorphous. The X-ray powder diffraction patterns of compounds III–V are characterized by intrinsic sets of interplanar distances and relative intensities (Table 2), indicating their individual character (Table 1).

Compounds I and II are nonelectrolytes, whereas compounds III–V are three-ion electrolytes, which is explained by the presence of two potassium cations in their molecules (Table 1).

According to the thermogravimetric data, the inner spheres of complexes I and IV contain coordinated water molecules, because their removal occurs at rather high temperature (~150°C) and is accompanied by weight loss.

The composition of the inner sphere in compounds **I–V** was determined by a set of physicochemical methods.

Comparison of the IR spectra of the ligands and complexes I–V (Table 3) reveals a low-frequency shift in the region of v(C=N) vibrations, indicating that the azomethine nitrogen atom is involved in coordination with M^{II}. This was also confirmed by the appearance of

the v(M-N) bands in the spectra of the complexes (Table 3).

The oxygen atoms of the hydroxy groups can presumably be involved in the formation of the coordination polyhedron of complexes **I** and **II**, which should result in a change in their vibrational bands. In fact, the band of stretching vibrations of the OH group of 1,8,2,4-ahKsHs'n is retained and the band of benzoin disappears in the IR spectra of compounds **I** and **II**. In this case, a band appears belonging to bending vibrations of the oxo bridge (710 cm⁻¹) between the ions of the corresponding M^{II}.

The IR spectra of complexes III–V contain no band of stretching vibrations of the OH group of 1,8,2,4ahKsHs'n at 3380 cm⁻¹ and, hence, L² in the monodeprotonated form is coordinated in compounds III–V. The band of stretching vibrations of the hydroxy group of the aldehyde is retained (Table 3). Note that the bands of stretching vibrations of the sulfo groups in the IR spectra of complexes III–V remain unchanged, whereas, in compounds I and II, they are shifted as compared to those for L² and L¹, respectively (Table 3). The bands of stretching and bending vibrations of the coordinated water molecules (Table 3) [11] are observed along with those observed in the IR spectra of complexes I and IV, which agrees with the thermogravimetric data.

Analysis and comparison with the published data [12-14] of the diffuse reflectance spectra of samples **III–V** and their magnetic moments [15] made it possible to establish the coordination polyhedron of these complexes: tetrahedral for **III**, octahedral for **IV**, and planar square-planar for **V**.

The dimeric structure of these complexes was assumed based on the data of the diffuse reflectance spectra (Table 4) and underestimated μ_{eff} values for complexes I and II as compared to the values characteristic of the octahedral environment of Ni^{II} (I) and tetrahedral environment of Co^{II} (II). This was confirmed by the EXAFS spectroscopic study of complex II.

The XANES (near edge fine structure) of the Co *K*-edge X-ray absorption spectrum and the correspond-

Compound	ν(OH)	v(C=N)	v(SO ₂)	ν(C–O)	ν(M–N)	v(M-O)
L ¹ [5]	3420, 3380	1600	1240, 1030	1210		
I *	3380	1560	1185	1180	580	460
Π	3380	1550	1185	1190	580	450
L^2	3450, 3380	1600	1240, 1030	1200		
III	3450	1575	1240, 1030	1200	600	480
IV*	3450	1560	1240, 1030	1200	603	420
V	3450	1550	1240, 1030	1200	600	460

Table 3. Assignment of some vibrational frequencies (cm^{-1}) in the IR absorption spectra of the ligands and complexes I–V

* $v(OH)_{H_2O} = 3300 \text{ cm}^{-1} (\mathbf{I}, \mathbf{IV}); \ \delta(H_2O) = 1620 (\mathbf{I}), 1618 \text{ cm}^{-1} (\mathbf{IV}).$

Compound		$\mu_{eff}, \mu B$		
Compound	ν ₁	v ₂	ν ₃	(T = 293 K)
	$^{3}A_{2g} \longrightarrow ^{3}T_{2g}(F)$	${}^{3}A_{2g} \longrightarrow {}^{3}T_{1g}(F)$	$^{3}A_{2g} \longrightarrow ^{3}T_{1g}(P)$	
Ι	9200	19600	26300	2.32
	${}^{4}A_{2} \longrightarrow {}^{4}T_{2}$	${}^{4}A_{2} \longrightarrow {}^{4}T_{1}(F)$	${}^{4}A_{2} \longrightarrow {}^{4}T_{1}(P)$	
II	4200	9300	18230	3.58
	$^{3}T_{1}(F) \longrightarrow ^{3}T_{2}$	$^{3}T_{1}(F) \longrightarrow ^{3}A_{2}$	${}^{3}\mathrm{T}_{1}\left(\mathrm{F}\right)^{3}\mathrm{T}_{1}\left(\mathrm{P}\right)$	
III		8930	15540	3.37
	${}^{4}\mathrm{T}_{1\mathrm{g}}\left(\mathrm{F}\right) \longrightarrow {}^{4}\mathrm{T}_{2\mathrm{g}}\left(\mathrm{F}\right)$	${}^{4}\mathrm{T}_{1\mathrm{g}}\left(\mathrm{F}\right) \longrightarrow {}^{4}\mathrm{A}_{2\mathrm{g}}$	${}^{4}\mathrm{T}_{1\mathrm{g}}\left(\mathrm{F}\right) \longrightarrow {}^{4}\mathrm{T}_{1\mathrm{g}}\left(\mathrm{F}\right)$	
IV	8000	18395	19800	5.13
		$^{2}B_{1g} \longrightarrow ^{2}A_{1g}$	$^{2}B_{1g} \longrightarrow ^{2}E_{g}$	
V		16660	19980	1.82

Table 4. Energies of electron transitions and effective magnetic moments of complexes I–V

ing first derivative of the spectrum for complex II are shown in Fig. 1. The XANES spectrum of complex II has pre-edge peak A at 7703 eV, shoulder B at 7709 eV, and major maximum C (white line) at 7117 eV. Preedge peak A corresponds to the electron transitions $1s \longrightarrow 3d$, and its intensity is determined by the degree of noncentrosymmetry of the coordination polyhedron of the metal ion [16]. Specific features of edges B and C are determined by electron transitions $1s \longrightarrow 4s$, $4p + L^1$ of the orbital of the ligands, respectively. This pattern of the XANES spectra is characteristic of a distorted tetrahedral coordination of a metal atom in a complex [17].



Fig. 1. The Co *K*-edge XANES spectrum (solid line) and its first derivative (dotted line) for complex **II**.

Figure 2 shows the Fourier transform magnitude (FTM) of the Co K-edge EXAFS spectrum of complex II along with the theoretical FTM obtained by nonlinear fitting of the parameters for two coordination spheres. The inset in Fig. 2 shows the experimental and theoretical EXAFS spectra isolated using the Fourier filtration from the total spectrum by the Kaiser-Bessel window in the range r = 0.9-3.1 Å. The FTM has two major peaks at r = 1.55 and 2.74 Å. The first peak unambiguously corresponds to the nearest coordination sphere consisting of the light N and O atoms. The coordination sphere corresponding to the second peak at high r values can contain both light atoms of the ligands (O, N) and the Co atoms (based on the assumption of the dimeric structure of the molecule). The following procedure was used to confirm the above assumption. The experimental FTM is obtained by the Fourier transformation of the EXAFS oscillations that are formed due to diffraction on all coordination spheres surrounding the absorbing atom, and the reflection amplitude depends on the type of the atom. Photoelectrons with a low value of the wave vector ($k < 6 \text{ Å}^{-1}$) correspond to the predominant scattering on the light atoms, and at $k > 6 \text{ Å}^{-1}$ the main contribution to the EXAFS spectrum is made by the scattering on the heavy atoms. Therefore, varying the integration limits during the Fourier transformation of the EXAFS spectrum one can separate the contributions of scattering on light and metal atoms. This procedure was performed for the EXAFS spectrum of complex II, the lower integration limit being shifted from k = 2.2 to 6 Å⁻¹.

Figure 3 clearly shows that the relative amplitude of the peak at r = 2.74 Å increases compared to that of the main peak (r = 1.55 Å) with an increase in the lower integration limit from k = 2.2 to 6 Å⁻¹. This presumably confirms that the peak is due to the scattering on the coordination sphere containing metal atoms.



Fig. 2. FTM of the Co *K*-edge EXAFS spectrum for complex **II** (experiment is solid line, and theoretical data are circles). Inset: the experimental and theoretical EXAFS spectra isolated by the Fourier filtration from the total spectrum using the Kaiser–Bessel window in the range r = 0.9-3.1 Å.



Fig. 3. FTM of complex **II** for the integration range of the EXAFS function (dotted line) from 2.2 to 14 Å⁻¹ and (solid line) from 6 to 14 Å⁻¹.

The quantitative characteristics for two isolated coordination spheres are given in Table 5. The theoretical phases and scattering amplitudes calculated for the dimeric complex [9] with a similar structure of the coordination core were used in fitting of the structural parameters. The best fitting was obtained for a model of the dimeric molecule in which the cobalt ions have the first coordination sphere composed of four O/N atoms at the average distance R = 2.09 Å and the coordination sphere with

the radius R = 3.12 Å containing one cobalt atom. Taking into account the XANES data, we can assert that the cobalt ion has a distorted tetrahedral coordination. The Co-Co distance (3.12 Å) obtained from the EXAFS data is characteristic of dimeric cobalt complexes with oxo bridges. Selected R values (Co-Co) for different types of oxo bridges taken from the CSD database are shown in Fig. 4. It is well seen that complex II has the Co-OR-Co bridge similar to that described in [18, 19].

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Table 5. Structural data for complex **II** obtained by fitting the EXAFS data (*R* are interatomic distances, σ^2 is the Debye–Waller factor, and *Q* is the function of fitting quality)

Compound	Coordination number	<i>R</i> , Å	σ^2 , Å ²	Atom	Q, %
II	4	2.09	0.0059	O/N	9.0
	1	3.12	0.0039	Со	

According to the aforesaid, complexes I and II have dimeric structures



Analysis of the ESR spectrum of complex V shows that the hyperfine structure lines are well resolved only in the parallel orientation, whereas a single unresolved



Fig. 4. Dependence of the *R*(Co–Co) distance in the dimeric cobalt complexes on the type of the oxo bridge.

line $(g_{\perp} = 2.058)$ is observed in the perpendicular orientation. The parameters of the ESR spectrum, calculated using a published program [20], have the values $g_{\parallel} =$ 2.235 and $A_{\parallel} = 170.0 \times 10^{-4}$ cm⁻¹, which, according to literature [21] and our [1, 5] data, corresponding to complexes with the CuO₂N₂ environment. This agrees with the above results of other investigation methods.

Finally, the following structures were proposed for complexes **III–V**:



Our findings show that Co(II) and Ni(II), unlike Cu(II), form with L^1 binuclear complexes with oxo bridges in which one of the sulfo groups of the ligand is additionally bound to the central ion. In the case of L^2 , the same bidentate coordination of the ligand in the monodeprotonated form through the azomethine nitrogen atom and the oxygen atom of the deprotonated OH group of 1,8,2,4-ahKsHs'n occurs in compounds **III–V**.

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