## **COORDINATION** COMPOUNDS =

# **Copper(II) Complexes with Condensation Products** of Aminonaphthalene and Benzoin Derivatives

L. S. Skorokhod<sup>a</sup>, I. I. Seifullina<sup>a</sup>, V. G. Vlasenko<sup>b</sup>, I. V. Pirog<sup>b</sup>, V. V. Minin<sup>c</sup>, V. E. Kuz'min<sup>d</sup>, and L. N. Ognichenko<sup>d</sup>

<sup>a</sup> Mechnikov National University, Dvoryanskaya ul. 2, Odessa, 65026 Ukraine

<sup>b</sup> Research Institute of Physics, Rostov State University, pr. Stachki 194, Rostov-on-Don, 344090 Russia

<sup>c</sup> Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences,

Leninskii pr. 31, Moscow, 119991 Russia

<sup>d</sup> Bogatskii Physicochemical Institute, National Academy of Sciences of Ukraine,

Lustdorfskaya doroga 86, Odessa, 65080 Ukraine

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Abstract—New copper(II) complexes with the products of condensation of benzoin with monopotassium 1-amino-8-hydroxy-2,4-naphthalenedisulfonate and 1,8-diaminonaphthalene (complexes I and II, respectively) have been synthesized. The compounds are identified and characterized by elemental analysis; X-ray powder diffraction; thermogravimetry, and electric conductivity data; magnetic susceptibility measurements; and IR, EPR, diffuse reflectance; EXAFS spectroscopy. The geometry and stability of the stereo isomers of complex I are theoretically studied with the use of the molecular mechanics method and semiempirical quantum-chemical calculations. The compositions of the inner spheres of complexes I and II and their coordination polyhedra—a distorted planar square (I) and a distorted octahedron (II)—are determined using different physicochemical methods.

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Previously [1, 2], we synthesized and studied Co(II) and Ni(II) complexes with the condensation products of aminonaphthalene and  $\alpha$ -hydroxy- $\alpha$ -phenylacetophenone (benzoin) derivatives. The complexes were synthesized by three methods: either (1) the above condensation products (ligands) or (2) the complexes with 1-amino-8-hydroxy-2,4-naphthalenedisulfonic acid were preliminarily prepared or (3) a template reaction on the Co(II) or Ni(II) matrix was carried out.

Similar copper(II) complexes were prepared only by the second method and characterized by different physicochemical methods, including EPR [3].

In this paper, we report on the synthesis and the results of physicochemical study of new Cu(II) complexes with a Schiff base (L<sup>1</sup>) and of a complex isolated from the  $CuCl_2-1,8$ -diaminonaphthalene-benzoin system.

### **EXPERIMENTAL**

Synthesis of a Schiff base (L<sup>1</sup>). Anhydrous sodium acetate (8.2 g, 0.10 mol) was added to a mixture of methanol solutions of 10.71 g (0.03 mol) of monopotassium 1-amino-8-hydroxy-2,4-naphthalenedisulfonate (1,8,2,4-ahKsHs'n) in 80 ml and 6.36 g (0.03 mol) of benzoin in 40 ml. The resulting solution was refluxed on a water bath for 1 h. The precipitate

formed was filtered off, washed with ethanol, and dried at room temperature over anhydrous calcium chloride to constant weight. The target product is a dark claret compound, mp 164°C.

For  $C_{24}H_{18}KNO_8S_2$  anal. calcd. (%): C, 52.27; H, 3.27; N, 2.54; S, 11.62; K, 7.08. Found (%) C, 52.81; H, 3.11; N, 2.59; S, 11.31; K, 7.29.



**Synthesis of complex I.** A reaction mixture containing 1.65 g (0.003 mol) of  $L^1$  (a solvent was a mixture of 50 ml of ethanol and 70 ml of methanol) and 0.51 g (0.003 mol) of copper(II) chloride in 15 ml of ethanol was refluxed on a water bath for 1 h. The precipitate formed after cooling the solution was worked up as described for  $L^1$ . The target product is a grey-green

compound insoluble in water and soluble in DMF and DMSO. The compound melts at 238°C and decomposes at 300°C.

For  $C_{48}H_{36}Cl_2K_2N_2O_{16}S_4Cu$  anal. calcd. (%): C, 46.58; H, 2.91; N, 2.26; Cl, 5.74; K, 6.31; S, 10.35; Cu, 5.14. Found (%): C, 46.02; H, 2.99; N, 2.00, Cl, 5.75; K, 6.03; S, 10.11; Cu, 5.26.

Synthesis of complex II. Hot ethanol solutions of 1.58 g (0.01 mol) of 1,8-diaminonaphthalene (1,8-aa'n) in 50 ml and 4.34 g (0.02 mol) of benzoin in 30 ml were poured together and refluxed for 1 h on a water bath. The resulting precipitate was worked up as described for  $L^1$  and complex I. Complex II is a dark brown crystalline substance soluble in DMF and DMSO and insoluble in water. Complex II decomposes above 250°C.

For C<sub>24</sub>H<sub>23</sub>ClN<sub>2</sub>O<sub>3</sub>Cu anal. calcd. (%): C, 59.26; H, 4.73; N, 5.76; Cl, 7.30; Cu, 13.07. Found (%): C, 59.51; H, 4.31; N, 5.59; Cl, 7.01; Cu, 12.89.

The copper content was determined by the X-ray fluorescence method on a SPARK-1 spectrometer (Cu radiation, 12 kV, 10 mA) at a counting rate of 400 counts/s. The potassium content was determined by the flame photometry method.

Thermogravimetric analysis was carried out on a Paulik-Paulik-Erdey Q derivatograph in air. Samples were heated at a rate of 10 K/min in the range 20– $500^{\circ}$ C.  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> was used as the reference.

X-ray powder diffraction patterns were recorded on a DRON-3 diffractometer ( $CuK_{\alpha}$  radiation, nickel filter). Interplanar spacings were determined using the tables [4].

The diffuse reflectance (DR) spectra (5000– 27000 cm<sup>-1</sup>) were recorded on a Perkin-Elmer Lambda 9 UV VIS NIR spectrophotometer. MgO was the reference. The IR absorption spectra were recorded as KBr pellets on a Specord IR-75 spectrophotometer in the range 400–4000 cm<sup>-1</sup>. Magnetic susceptibility was determined at 293 K by the Gouy method. Hg[Co(NCS)<sub>4</sub>] was used as a reference for calibration.

For calculation of molar conductivity, the ohmic resistance of  $1 \times 10^{-3}$  M solutions of L<sup>1</sup> and compounds I and II in DMF placed in an Arrhenius flask was measured using an E7-8 digital resistance meter in the range 0–10 m $\Omega$ . The EPR spectra were recorded on a Radiopan SE/X-2542 radiospectrometer operating at 9.4 GHz. The magnetic field sweep was calibrated with a nuclear magnetometer. Diphenylpicrylhydrazyl (DPPH) was used as a reference for the determination of *g* values. Solutions in DMF with the concentration of the complexes of  $1 \times 10^{-2}$  mol/l were studied. The room-temperature EPR spectra of these solutions showed a broad band caused by the Jahn–Teller effect [5]; therefore, all measurements were taken at 77 K.

Copper *K*-edge EXAFS spectra were recorded in the transmission mode on an EXAFS spectrometer at the Siberian Synchrotron Radiation Center (Novosibirsk).

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The energy of an electron beam used as the source of X-ray synchrotron radiation was 2 GeV at an average beam current of 80 mA. X-ray radiation was monochromatized by a Si(111) double-crystal monochromator. The incident and transmitted X-ray radiation intensities were measured by argon-filled ionization chambers.

Samples for EXAFS experiments were thoroughly blended with Apiezon and placed between thin lavsan films. The sample thickness was selected so that the transmitted X-ray radiation intensity was 2.5–3.0 times as low as the incident beam intensity. After standard procedures for background removal, normalization to the *K*-edge jump, and extraction of atomic absorption  $\mu_0$  [6], the resulting EXAFS ( $\chi$ ) spectra were Fourier transformed in the range of photoelectron wave vectors *k* from 2.6 to 13.3 A<sup>-1</sup> with the weighting function n = 2. The threshold ionization energy  $E_0$  was chosen based on the maximum of the first derivative of the *K*edge and, then, varied in the fitting procedure.

The Fourier transform magnitude (FTM) obtained by Fourier transformation of the EXAFS spectrum represents the radial distribution function of neighboring atoms around the absorbing metal atom. The abscissas of the maxima of the FTM peaks (r) are related to the radii of coordination spheres (R) by the formula r = R - R $\alpha$ , where  $\alpha$  is the linear part of the phase shift. The FTM peak amplitudes are proportional to the coordination numbers (N). The precise structural parameters of the nearest environment of metal atoms were determined by nonlinear fitting of the calculated EXAFS signal to the FTM extracted from the full EXAFS spectrum by Fourier filtration. This nonlinear fitting was performed with the IFFEFIT-1.2.5 program package [7]. The scattering phase shifts and amplitudes of a photoelectron wave, which were required for constructing a model spectrum, were calculated with the FEFF7 program [8]. Metal complexes with similar structures characterized by X-ray crystallography were used as model compounds.

The goodness-of-fit function Q, which was minimized when finding the structural parameters of the nearest environment, was calculated by the formula

$$Q(\%) = \frac{\Sigma[k\chi_{\exp}(k) - k\chi_{\tanh}(k)]^2}{\Sigma[k\chi_{\exp}(k)]^2} \times 100.$$

The geometries and relative stabilities of the stereo isomers of complex I were calculated by both the molecular mechanics force field (MMFF) method [9] and the semiempirical  $PM_3$  method [10].

#### **RESULTS AND DISCUSSION**

The condensation of 1,8,2,4-ahKsHs'n with benzoin yielded ligand  $L^1$ . Complex I was synthesized on the basis of this ligand. Complex II was obtained by the

Compound	v(OH)	v(C=N)	$v(SO_2)$	v(C–O)	v(Cu–N)	v(Cu–O)
L <sup>1</sup>	3420, 3380	1600	1240, 1030	1210		
Ι	3530, 3200	1570	1240, 1030	1180	595	252
<b>II</b> *	3600-3400	1555		1170	587	490

**Table 1.** Assignment of selected vibrational frequencies  $(cm^{-1})$  in the IR absorption spectra of benzoin, 1,8-aa'n, and complexes I and II

Note: 1,8-aa'n:  $v(NH)_2$ ) = 3400 cm<sup>-1</sup> (3300 cm<sup>-1</sup> in **II**),  $\delta(NH_2)$  = 1630 cm<sup>-1</sup> (1609 cm<sup>-1</sup> in **II**). Benzoin: v(OH) = 3420, 3380 cm<sup>-1</sup>; v(C=O) = 1680 cm<sup>-1</sup>; v(C=O) = 1210 cm<sup>-1</sup>.

\*  $\delta(H_2O) = 1620 \text{ cm}^{-1}$ .

reaction of  $\text{CuCl}_2$  with 1,8-aa'n and benzoin. The  $\text{Cu}^{2+}$ : ligand molar ratio is 1 : 2 in I and 1 : 1 in II. The individuality of I and II was demonstrated by X-ray powder diffraction data.

Compounds L<sup>1</sup> and **I** are X-ray amorphous. The X-ray powder diffraction pattern of **II** is characterized by an individual set of interplanar spacings and relative intensities: *d*, Å ( $I/I_0$ , %): 2.61 (28), 2.79 (100), 3.07 (41), 3.14 (23), 6.85 (23), 8.28 (23), 8.94 (55), and 9.81 (32).

The molar conductivity of **I** is  $132.4 \ \Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$ , which corresponds to a three-ion electrolyte [11] and is consistent with the presence of two chloride ions in the outer sphere of the complex as found by chemical analysis. In contrast to **I**, compound **II** is a nonelectrolyte ( $\lambda = 20.1 \ \Omega^{-1} \text{ cm}^2 \text{ mol}^{-1}$ ), which points to the innersphere location of the chloride ion. The inner sphere of **II** also involves two water molecules, since their removal takes place at a rather high temperature (~140°C) and is accompanied by a corresponding weight loss.

The coordination modes of the ligands were determined from IR spectroscopy data. Comparison of the IR spectra of  $L^1$  and complex I (Table 1) shows a lowfrequency shift of the v(C=N) absorption band, which is evidence that the azomethine nitrogen atom is coordinated to Cu<sup>2+</sup>. The copper coordination polyhedron in **I** is also formed by the oxygen atoms of the hydroxy groups of the benzoin and naphthol moieties, which should lead to a change in their vibrational frequencies. Indeed, the IR spectrum of I (Table 1) shows that the complex has nonequivalent OH groups (coordinated to Cu<sup>2+</sup> and free). The fact that the sulfo group vibration frequencies in the spectrum of I remain the same as in the spectrum of  $L^1$  allows us to conclude that these groups do not form bonds to  $Cu^{2+}$  (Table 1). Thus,  $L^1$  in complex I acts as a bidentate ligand.

The DR spectrum of a polycrystalline sample of **I** shows the  ${}^{2}B_{1g} \longrightarrow {}^{2}A_{1g}$  (16700 cm<sup>-1</sup>) and  ${}^{2}B_{2g} \longrightarrow {}^{2}E_{g}$  (19500 cm<sup>-1</sup>) transitions typical of the square-planar

environment of Cu<sup>2+</sup>. This is consistent with the effective magnetic moment of I:  $\mu = 1.860\mu_B$  [12].

The EPR spectrum of **I** shows that hyperfine structure (HFS) lines are well resolved only in parallel orientation, while only one unresolved line is observed in perpendicular orientation. The EPR parameters determined according to [13] are  $g_{\parallel} = 2.240$  and  $A_{\parallel} = 172.0 \times 10^{-4}$  cm<sup>-1</sup> ( $g_{\perp} = 2.033$ ). According to the available data, these parameters can be assigned to a complex with the CuO<sub>2</sub>N<sub>2</sub> coordination core [14]; i.e., each copper ion is coordinated to two ligand molecules through the azomethine nitrogen atom and hydroxyl oxygen atom.

The set of donor atoms in **I** determined by EPR is in agreement with its composition and the coordination mode of the ligands suggested above.

The IR spectrum of **II** shows a broad band at 3600-3400 cm<sup>-1</sup>, and the bands due to  $\delta(H_2O)$ ,  $\nu(NH_2)$ , v(C=N), and  $v(NH_2)$  (Table 1). The positions of the last three bands indicate that the corresponding groups are bonded to copper(II) in complex II. One more bond to Cu(II) is through the oxygen atom of the deprotonated hydroxy group of the benzoin moiety, which is demonstrated by the corresponding shift of the v(C-O) band in the IR spectrum of **II** compared to its position in the spectrum of benzoin (Table 1). Two water molecules and a chloride ion increase the coordination number of copper(II) to six. This is consistent with elemental analysis and thermogravimetry data, the value of the effective magnetic moment ( $\mu = 2.00\mu_B$ ), and the presence of one transition  $E_g \longrightarrow T_{2g}$  (~14000 cm<sup>-1</sup>) in the DR spectrum typical of the octahedral coordination of the Cu<sup>2+</sup> ion [15].

Additional information on the structures of the coordination cores in I and II was obtained by EXAFS studies.

Figure 1 shows the experimental copper X-ray absorption near-edge structure (XANES) spectra and their first derivatives.

Analysis of the XANES spectra of I and II shows that they have very weak features in the form of preedge peaks at 8982 eV and a shoulder at 8990 eV, which are caused by the electronic transitions  $1s \rightarrow$ 



Fig. 1. Normalized copper XANES spectra of compounds (1) I and (2) II and (1', 2') their first derivatives.

3d(metal) and  $1s \longrightarrow 4s$  and  $1s \longrightarrow 4p(\text{ligand})$ , respectively. The intensities of these features in the XANES spectra of 3d metals are related to the geometry of the coordination core [16]. The XANES spectrum of I has two main maxima at 8990 and 8995 eV. This XANES pattern is characteristic of a distorted planar coordination of a metal atom in a complex. When symmetry is lowered, the degeneracy of the 4p atomic orbital of the metal is lifted, which is manifested in the XANES spectrum as a near-edge absorption peak. The copper XANES spectrum of **II** differs considerably from the XANES spectrum of I. First of all, the maximum at 8995 eV has a low amplitude. This can be explained by a lower degree of s-p mixing, i.e., by a higher symmetry of the coordination core, which is most likely a distorted octahedron.

Figures 2 and 3 show the FTMs of CuK-edge EXAFS functions for complexes I and II. Table 2 presents the structural parameters for the nearest atomic environment of the copper ion in complexes I and II. In I, the first coordination sphere is composed of the four atoms (N/O) of the ligands at a distance of 2.00 Å, which is typical of the copper complexes with a planar coordination. The nearest atomic environment of the Cu ions in II was modeled assuming an octahedral environment. The first coordination sphere in this model contains one chlorine atom, two oxygen atoms of the coordinated water molecules, and the nitrogen and oxygen atoms of the ligands, their number being varied in the course of fitting. As follows from Table 2, the coordination of three N/O atoms with the Debye-Waller factors typical of the given coordination sphere leads to a very low residual factor Q, which serves as numerical criterion for the correctness of the model used. The distances to the oxygen atoms of the coordinated water molecules are considerably longer than the



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Fig. 2. FTM of CuK-edge EXAFS data for complex I.

Cu–N/O(ligand) distances, which is typical of octahedral copper complexes due to the Jahn–Teller effect. The second and subsequent coordination spheres containing a large number of aromatic carbon atoms with a large dispersion of distances to the absorbing copper atom were not considered.

Our findings allow us to suggest the following structure for complex **II**:



The data obtained for I give no way of unambigu-

**Table 2.** Structural characteristics of the nearest atomic environment of the copper atoms in **I** and **II** obtained by fitting the EXAFS data (*N* is the coordination number, *R* is the interactomic distance,  $\sigma^2$  is the Debye–Waller factor, *Q* is the residual function)

Com- pound	Ν	<i>R</i> , Å	$\sigma^2$ , Å <sup>2</sup>	Atom	<i>Q</i> , %
Ι	4	2.00	0.0053	O/N	1.8
Π	2.8	1.96	0.0039	O/N	0.3
	2.0	2.13	0.0064	$O(H_2O)$	
	1	2.20	0.0042	Cl	



Fig. 3. FTM of CuK-edge EXAFS data for complex II.

ously deciding which of the hydroxy groups (benzoin or naphthol) is bonded to the copper(II) atom; therefore, we consider all possible structures of this complex, taking into account not only different variants of coordination of these groups but also *cis* and *trans* isomerism.





The calculated energies and geometrical characteristics of the coordination core are summarized in Table 3. Both approaches predict the same most stable stereo isomers of I: *cis*-3, *trans*-2, and *trans*-3.

In the *trans* isomers, the coordination core is a somewhat pyramidally distorted planar square. In the *cis* isomers, the coordination core is somewhat tetrahedrally distorted. The distances between the  $Cu^{2+}$  ion and the donor atoms of the ligands in these isomers are 1.92 (PM3) and 2.19 Å (MMFF). These values are close to the experimental value 1.97 Å obtained from the EXAFS data.

Our findings show that the *trans*-3 isomer of complex **I** is the most probable. Both calculation methods, molecular mechanics and quantum-chemical calculations, predict that this isomer is the most stable. It has five-membered chelating rings. The Cu–O bonds are slightly out of the plane of the coordination core so that it is somewhat pyramidally distorted. The aromatic nuclei form a cyclic hydrophobic shell around the coordination core, which is protected from above and below by the  $-SO_3K$  groups.

		bond angles, deg	(1)CuN(2)	107.8	124.1	156.3	97.7	92.5	90.1			(1)CuN(2)	180.0	176.8	128.8	96.8	102.8	107.7
			D(2)CuN(1) O	164.3	123.9	101.7	86.3	87.6	97.4		bond angles, deg	D(2)CuN(1) O	176.4	170.3	157.1	105.5	95.8	100.0
			O(2)CuN(2)	90.0	80.3	88.1	88.1	90.3	90.8			O(2)CuN(2)	80.5	77.2	76.1	82.3	78.8	76.9
			N(1)CuN(2)	103.3	155.8	102.3	169.6	177.6	128.1	-		N(1)CuN(2)	103.0	107.2	124.9	171.7	174.5	175.8
lculations			O(1)CuO(2)	86.4	60.3	110.7	174.1	162.9	170.7			O(1)CuO(2)	99.4	99.8	87.1	178.9	178.2	169.7
	alculations		O(1)CuN(1)	97.2	73.2	88.3	87.8	89.9	89.3	ulations		O(1)CuN(1)	77.1	75.6	72.7	75.3	82.6	75.9
	HyperChem ca	average bond lengths, Å		1.92	2.01	1.91	1.94	1.87	1.89	Spartan calc	average bond lengths, Å		2.24	2.24	2.13	2.22	2.12	2.16
			Cu–N(2)	1.91	1.90	1.89	1.94	1.86	1.86			Cu-N(2)	2.24	2.36	1.92	2.18	2.11	2.16
		bond lengths, Å	Cu-O(2)	1.90	2.07	2.00	1.95	1.89	1.89		bond lengths, Å	Cu-O(2)	2.17	2.12	2.43	2.10	2.12	2.12
			Cu-N(1)	1.90	1.89	1.89	1.92	1.89	1.97			Cu-N(1)	2.39	2.23	2.16	2.50	2.10	2.09
is- trans			Cu-O(1)	1.97	2.17	1.86	1.94	1.85	1.86			Cu-O(1)	2.17	2.25	2.01	2.12	2.17	2.25
		PM3	E, kcal/mol	-11630	-11639	-11644	-11593	-11646	-11653		MMFF	<i>E</i> , kcal/mol	469	356	321	483	337	236
2				cis-1	cis-2	cis-3	trans-1	trans-2	trans-3				cis-1	cis-2	cis-3	trans-1	trans-2	trans-3

Table 3. Results of the calculations of the energy and geometry of the coordination core of complex I

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