

Molecular Design of New Magnetically Active Copper Complexes with Heteroaromatic Schiff Bases and Azo Compounds

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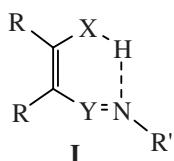
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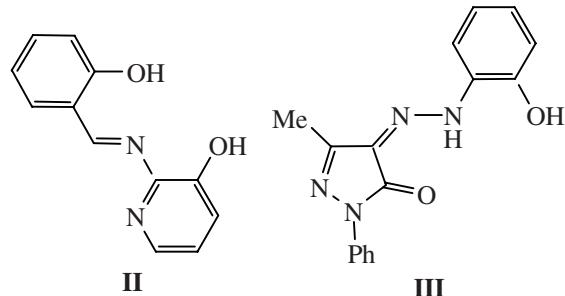
Abstract—Copper chelates with tridentate ligands containing pyridine or pyrazole ring at the azomethine or azo fragment were synthesized by chemical electrochemical methods, and their structure was characterized by the EXAFS spectra. Thermal magnetochemical analysis of the complexes revealed antiferromagnetic exchange interaction in all complexes. The exchange interaction parameter of the complex containing an *N*-tosylamino group in the *ortho* position with respect to the azomethine group is much lesser than that of the corresponding complex having an oxygen atom in the same position. The copper chelate derived from azopyrazole ligand shows low-temperature ferromagnetic phase transition.

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Molecular design principles [1–5] are widely used in the creation of magnetically active metal complexes of Schiff bases and their analogs [6–10]. Such complexes are usually [4, 5] synthesized from ligands of general structure **I** where aldehyde (R) and amine (R') components, donor atoms X, and fragments Y are varied. In continuation of our previous studies [11–15] we synthesized and characterized tridentate heterocyclic ligands, Schiff base **II** and azo pyrazole **III**. Ligands **II** and **III** were used to obtain dinuclear complexes **IV** and **V**, and the structure and magnetic properties of the latter were examined.

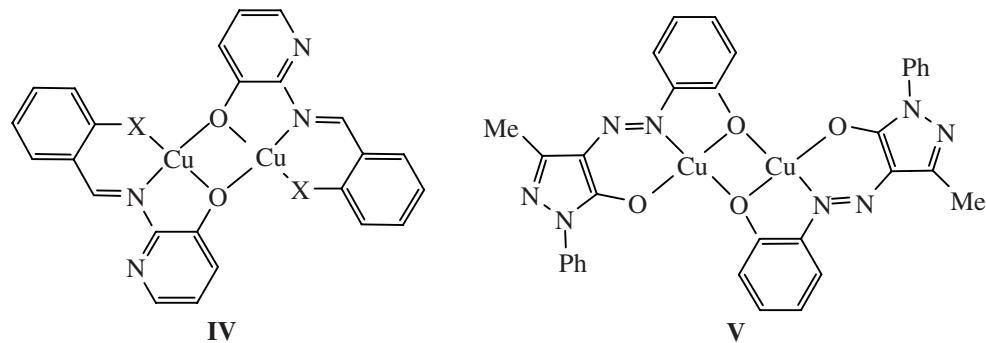


I, R = Alk, Ar, Ht; R' = Alk, Ar, Ht; X = NR², O, S, Se; Y = CR³, N; R² = H, Alk, Ar, Ts; R³ = H, Alk, Ar; Ts = p-MeC₆H₄SO₂.



Schiff base **II** exists mainly in the enol form [4, 5], while hydrazone tautomer **III** is typical of the azo pyrazole ligand [5, 16]. The corresponding coordination copper compounds have the composition Cu₂L₂ (where LH₂ is ligand **II** or **III**) and chelate structure with almost equivalent metal–ligand bonds, which excludes (cf. [5, 17, 18]) consideration of tautomeric anionic ligand systems. The chelate structure follows from the IR spectra of complexes **IV** and **V**, which lack OH and NH stretching vibration bands in the region 3100–3400 cm^{−1}; the frequency of stretching vibrations of the C=N group in the complexes

[†] Deceased.

**IV**, X = O (**a**); X = NTs (**b**).

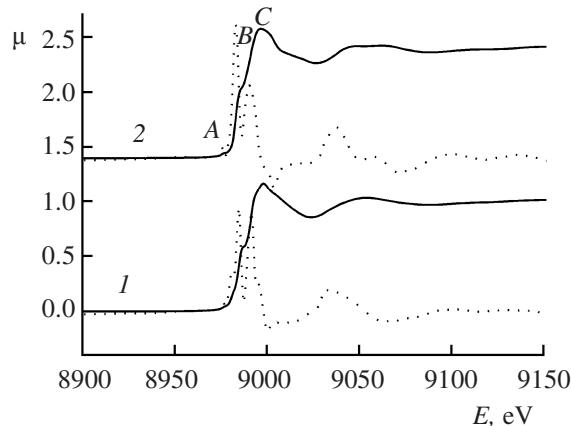
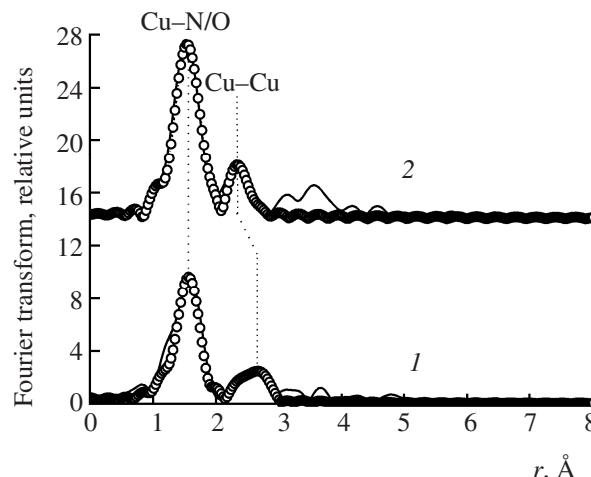
decreased by 10–15 cm⁻¹, while the Ph–O vibration frequency increases by 50 cm⁻¹, as compared to the corresponding free ligands [19, 20]. The complexes synthesized by chemical and electrochemical methods had identical spectral parameters.

The dinuclear structure and coordination mode were determined by analysis of the EXAFS spectra of complexes **IVb** and **V**. The CuK-edge XANES spectra of **V** and **IVb** (Fig. 1) displayed very weak near-edge peaks *A* at 8977.5 eV, a well resolved shoulder *B* at 8987 eV, and the main absorption maximum *C* at 8998 eV. This pattern results from electron transitions $1s \rightarrow 3d$, $1s \rightarrow 4s$, and $1s \rightarrow 4p + \text{ligand}$ orbitals, respectively. The first derivatives of the CuK-edges for compounds **IVb** and **V** are characterized by distinctly split maxima, which is typical of planar coordination of copper ion.

Figure 2 shows Fourier transforms of the CuK-edge EXAFS spectra of **IVb** and **V**. Parameters of the

nearest environment of the copper ion in complexes **IVb** and **V** are collected in table. They were obtained by fitting the $\chi(k)$ values calculated for the corresponding model structures to the experimental EXAFS spectra. As model structure we used bis[4-(5-chloro-2-hydroxyphenylimino)pent-2-en-2-olato]dicopper(II) [21] with the distances Cu–O 1.94 and 1.95 Å and Cu–Cu 2.994 Å.

The Fourier transforms of both samples (Fig. 2) consist of the main peak at $r = 1.55$ Å and a peak with a lower amplitude at $r = 2.66$ Å (**IVb**) or 2.36 Å (**V**). The main peak corresponds to photoelectron scattering at the nearest coordination spheres of nitrogen and oxygen, whose radii were determined by fitting (see table). The radii of the first coordination sphere are typical of structurally related copper complexes. The second peak, in keeping with the proposed dinuclear structure, can be assigned to the Cu–Cu distance. It should be noted that the Cu–Cu distance in complex **V**

**Fig. 1.** CuK-edge XANES spectra (solid lines) and their first derivatives (dotted lines) of complexes (1) **IVb** and (2) **V**.**Fig. 2.** Fourier transforms (circled lines) of the CuK-edge EXAFS spectra (solid lines) of complexes (1) **IVb** and (2) **V**.

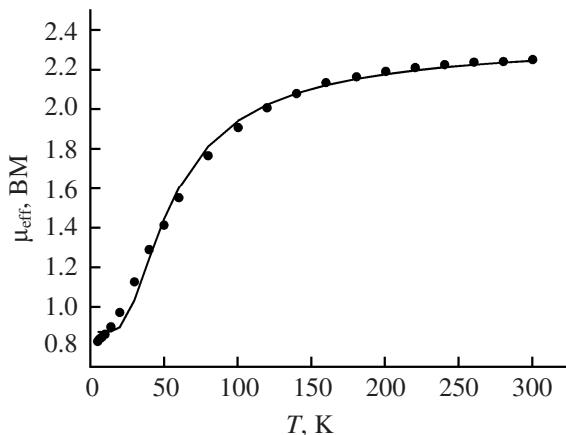


Fig. 3. Temperature dependence of the effective magnetic moment of complex IVa.

is shorter than in IVb by 0.26 Å. Thus the EXAFS spectra of copper complexes IVb and V confirmed their dimeric structure.

Magnetochemical studies revealed antiferromagnetic exchange interactions in complexes IV and V. Figures 3–5 show the experimental and calculated temperature dependences of the effective magnetic moments and magnetic susceptibilities of complexes IV and V. Assuming binuclear structure of the complexes, the dependences were simulated using the Bleany–Bowers equation for magnetic susceptibility of the dimer [7]:

$$\chi_{(\text{Cu–Cu})} = (N\beta^2 g^2 / 3kT) [1 + 1/3 \exp(-2J/kT)]^{-1} + N\alpha$$

with account taken of exchange interaction between dimers zJ' :

$$\chi' = \frac{\chi_{(\text{Cu–Cu})}}{1 - (2zJ'/Ng^2\beta^2)\chi_{(\text{Cu–Cu})}}$$

and possible impurity p of monomeric species with a spin $S = 1/2$:

Structural parameters determined by multisphere fitting of the EXAFS spectra (R stands for interatomic distances, N is the coordination number, σ^2 is the Debye–Waller factor, and Q is the goodness of fit function

Comp. no.	N	R , Å	σ^2 , Å ²	Atom	Q , %
IVb	4	1.93	0.0035	N	5.3
	1	2.96	0.0039	Cu	
V	5	1.94	0.0027	O/N	2.7
	1	2.70	0.0037	Cu	

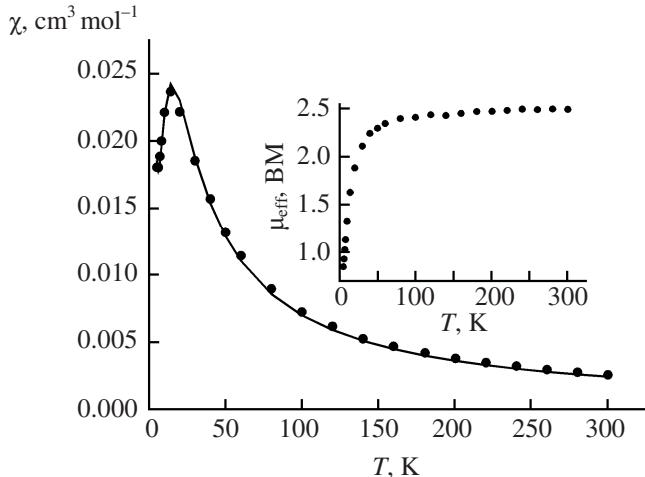


Fig. 4. Temperature dependence of the effective magnetic moment of complex IVb.

$$\chi = \chi' (1 - p) + [N\beta^2 g^2 S(S+1)/3k(T-\theta)]p.$$

Here, N , $N\alpha$, k , β , J , g , and θ are respectively the Avogadro number, van Vleck paramagnetism, Boltzmann constant, Bohr magneton, exchange interaction parameter, Landé g -factor, and Weiss constant. The calculated optimal parameters are given below.

Comp. no.	IVa	IVb	V
g	1.92	2.14	2.11
J , cm ⁻¹	-42	-9.7	-210
p	0.13	0.22	0.002

Complexes IV are characterized by antiferromagnetic exchange [22] which is considerably stronger in IVa ($J = -42$ cm⁻¹) than in IVb ($J = -9.7$ cm⁻¹). This means that replacement of the donor group X in IVa (X = O) by *N*-tosyl fragment (IVb, X = NTs) considerably weakens antiferromagnetic exchange interaction between the paramagnetic centers. Figure 5 shows that antiferromagnetic exchange between the paramagnetic centers is also characteristic of metal complex V. However, in the low-temperature region (~20 K), the complex undergoes ferromagnetic phase transition, and its unusual behavior attracts essential interest.

EXPERIMENTAL

The IR spectra were recorded on a Nicolet Impact-400 instrument from samples dispersed in mineral oil. The ¹H NMR spectra were measured on a Varian Unity-300 spectrometer at (300 MHz) using internal stabilization with respect to ²H in CDCl₃ or DMSO-*d*₆.

The CuK-edge X-ray absorption spectra were recorded in the transmission mode on an EXAFS spectrometer at the Structural Material Science Station, Kurchatov Synchrotron Center (Moscow, Russia). The energy of electron beam used as source of X-ray synchrotron radiation was 2.5 GeV at a current of 80–100 mA. The X-ray radiation was monochromatized using a two-crystal Si(111) monochromator. The spectra were processed by standard procedures for background removal, normalization by the magnitude of *K*-edge jump, and isolation of atomic absorption μ_0 [23]; the EXAFS (χ) spectra were then subjected to Fourier transform in the range of photoelectron wave vectors k from 2.6 to 13 Å⁻¹ with weight function k^2 . The threshold ionization energy E_0 was assumed to be equal to the maximum of the first derivative of *K*-edge and was then adjusted by fitting. The exact structural parameters of the nearest environment of copper atoms were determined by nonlinear fitting of parameters of the corresponding coordination spheres via comparison of the calculated EXAFS signal with that isolated from the total EXAFS spectrum by filtration of the Fourier transform. Nonlinear fitting was performed using IFFEFIT-1.2.5 software package [24]. The photoelectron scattering phases and amplitudes required for simulation of the EXAFS spectrum were calculated using FEFF7 program [25]; X-ray diffraction data for a single crystal of bis[4-(5-chloro-2-hydroxyphenylimino)pent-2-en-2-olato]dicopper(II) [21] ($C_{22}H_{20}Cl_2Cu_2N_2O_4$, TEKYIR [26]) were used as initial coordinates of atoms for the calculation of scattering phases and amplitudes and subsequent fitting. The goodness of fit function Q was calculated by the formula

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

Minimization of Q was performed while determining structural parameters of the nearest environment of copper ions.

Magnetic properties of complexes **IV** and **V** were studied on a Quantum Design SQUID magnetometer in the temperature range from 300 to 2 K; magnetic field strength 5 kOe. The effective magnetic moments at different temperatures were calculated by the formula

$$\mu_{\text{ef}}(T) = [(3k/N\beta^2)\chi T]^{1/2} \approx (8\chi T)^{1/2}.$$

Schiff base **II** was synthesized according to the procedure described in [22, 27] by reaction of 2-

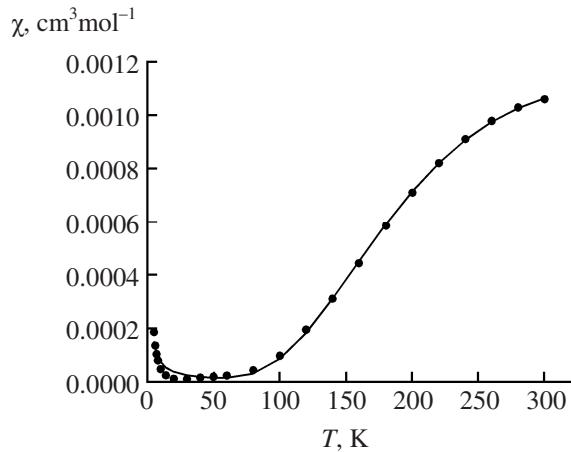


Fig. 5. Temperature dependence of the specific magnetic susceptibility of complex **V**.

aminopyridin-3-ol with 2-hydroxybenzaldehyde. Ligand **III** was synthesized as described for other azo pyrazole derivatives [16, 28], by diazo coupling of *o*-hydroxybenzenediazonium salt with 1-phenyl-3-methyl-4,5-dihydro-1*H*-pyrazol-5-one.

2-[(2-Hydroxyphenyl)methylidene]amino} pyridin-3-ol (II). Salicylaldehyde, 10 mmol, was added to a solution of 10 mmol of 2-aminopyridin-3-ol in 50 ml of ethanol, and the mixture was heated for 2 h under reflux. After cooling, the precipitate was filtered off and dried on exposure to air. Yellow-orange crystals, mp 168–170°C (decomp.) IR spectrum, ν , cm⁻¹: 3387 (OH), 1615 (C=N), 1274 (Ph-O). ¹H NMR spectrum ($DMSO-d_6$), δ , ppm: 6.86–7.93 m (7H, H_{arom}), 9.40 s (1H, CH=N), 9.91 s (1H, OH), 13.69 s (1H, OH). Found, %: C 67.38; H 4.61; N 13.18. $C_{12}H_{10}N_2O_2$. Calculated, %: C 67.28; H 4.71; N 13.08.

4-(2-Hydroxyphenylhydrazone)-3-methyl-1-phenyl-4,5-dihydro-1*H*-pyrazol-5-one (III). Finely powdered sodium nitrite, 22 mmol, was added in portions to a solution of 20 mmol of *o*-aminophenol in a mixture of 10 ml of acetic acid and 10 ml of methanol under stirring at 10°C so that to avoid evolution of nitrogen oxides. The mixture was kept on cooling for 30 min, and 20 ml of water was added in portions. The resulting diazonium salt solution was slowly added under stirring to a mixture of 20 mmol of 3-methyl-1-phenyl-4,5-dihydro-1*H*-pyrazol-5-one, 20 ml of acetic acid, and 30 ml of methanol, the mixture was kept for 10 h at room temperature and adjusted to pH 6–7 by adding a solution of sodium carbonate, and the precipitate was filtered off, washed with 4–6 portions of water, and dried in air. Yield 88%. mp 238–239°C

(from ethanol). IR spectrum, ν , cm^{-1} : 3100 (N—H), 1660 (C=O), 1621 (C=N), 1233 (Ph—O). ^1H NMR spectrum (CDCl_3), δ , ppm: 2.38 s (3H, CH_3), 6.93–7.97 m (9H, H_{arom}), 12.25 s (1H, OH), 13.59 s (1H, NH). Found, %: C 65.43; H 4.85; N 18.97. $\text{C}_{16}\text{H}_{14}\text{N}_4\text{O}_2$. Calculated, %: C 65.30; H 4.79; N 19.03.

Copper complexes **IV** and **V** were synthesized by direct reaction of copper(II) acetate with the corresponding ligands, as well as by electrochemical method (**IVa**, **V**) or template synthesis (**IVb**) [29–30].

Bis[2-{[2-hydroxyphenyl)methylidene]amino}pyridin-3-olato]dicopper(II) (IVa**).** A solution of 1 mmol of copper(II) acetate monohydrate in 20 ml of ethanol was added to a hot solution of 1 mmol of Schiff base **II** in 50 ml of the same solvent, and the mixture was heated for 4 h under reflux in an argon atmosphere. After cooling, the precipitate was filtered off, washed with hot ethanol (3×5 ml), and dried at 100°C under reduced pressure. Yield 75%, brown powder, mp > 250°C. IR spectrum, ν , cm^{-1} : 1605 (C=N), 1324 (Ph—O). Found, %: C 52.37; H 2.85; N 10.27. $\text{C}_{24}\text{H}_{16}\text{N}_4\text{O}_4\text{Cu}_2$. Calculated, %: C 52.27; H 2.92; N 10.16.

Bis[2-{[(2-tosylaminophenyl)methylidene]amino}pyridin-3-olato]dicopper(II) (IVb**).** A solution of 1 mmol of 2-aminopyridin-3-ol in 20 ml of ethanol was added to a hot solution of 1 mmol of 2-tosylaminobenzaldehyde [31] in 30 ml of ethanol, and the mixture was heated for 2 h under reflux in an argon atmosphere. A solution of 1 mmol of copper(II) acetate monohydrate in 20 ml of the same solvent was added, and the mixture was heated under reflux for 2 h. The mixture was cooled to room temperature, and the precipitate was filtered off, washed with boiling ethanol (3×5 ml), and dried at 150°C under reduced pressure. Yield 70%, brown powder, mp > 250°C. IR spectrum, ν , cm^{-1} : 1608 (C=N), 1290 ($\nu_{\text{as}}\text{SO}_2$), 1090 ($\nu_s\text{SO}_2$). Found, %: C 53.25; H 3.61; N 9.76. $\text{C}_{38}\text{H}_{30}\text{N}_6\text{O}_6\text{S}_2\text{Cu}_2$. Calculated, %: C 53.20; H 3.52; N 9.80.

Bis[4-(2-hydroxyphenyldiazeno)-3-methyl-1-phenyl-4,5-dihydro-1*H*-pyrazol-5-onato]dicopper(II) (V**).** A solution of 1 mmol of copper(II) acetate monohydrate in 20 ml of methanol was added under argon to a solution of 1 mmol of compound **III** in 5 ml of methanol, and the mixture was heated for 1 h on a water bath. The precipitate was filtered off, washed with hot methanol (3×10 ml), and dried at 150°C under reduced pressure. Yield 90%, brown powder, mp

> 250°C. IR spectrum, ν , cm^{-1} : 1593 (C=N), 1300 (Ph—O). Found, %: C 54.14; H 3.30; N 15.86. $\text{C}_{32}\text{H}_{12}\text{N}_4\text{O}_2\text{Cu}_2$. Calculated, %: C 54.01; H 3.40; N 15.76.

Electrochemical syntheses of complexes **IVa and **V**.** These syntheses were carried out according to the standard procedure [32] by reaction of compound **II** or **III**, respectively, with copper cations generated from the anode which dissolved during the electrolysis. The electrochemical cell may be represented as follows: Pt(–)/ $\text{CH}_3\text{CN}/\text{H}_2\text{L}$ (**II** or **III**)/Cu(+). The acetonitrile solution (25 ml) contained 1 mmol of ligand **II** or **III** and 0.01 g Et_4NClO_4 as supporting electrolyte. The syntheses were performed at a constant current of 40 mA and voltage of 15 V; reaction time 2 h. The precipitate was filtered off, washed with hot ethanol (3×10 ml), and dried at 150°C under reduced pressure.

Complex **IVa.** Yield 68%. IR spectrum, ν , cm^{-1} : 1606 (C=N), 1324 (Ph—O). Found, %: C 52.35; H 2.84; N 10.27.

Complex **V.** Yield 85%. IR spectrum, ν , cm^{-1} : 1590 (C=N), 1290 (Ph—O). Found, %: C 54.15; H 3.28; N 15.85.

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