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Synthesis and Magnetic Properties of New Binuclear Cu(II) Complexes with Tridentate Azomethine Ligands

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Abstract—New binuclear copper complexes of tridentate azomethine ligands with various combinations of N, O, and S donor centers were prepared by various procedures, including template synthesis. The magnetochemical data obtained for the range 2–300 K suggest the occurrence of antiferromagnetic coupling in most of these chelates. The only exception is the complex containing *N*-tosyl and *N*-ethyl donor fragments, in which the ferromagnetic exchange is observed.

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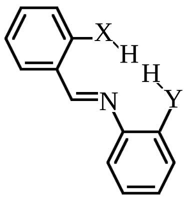
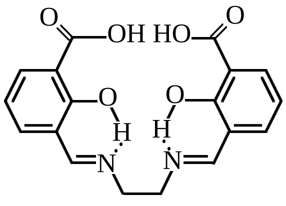
Proceeding with studies on the development of magnetoactive materials [1–10], among which a prominent place is occupied by Schiff base complexes, we prepared a series of copper complexes of tridentate Schiff bases **I** (LH_2) and examined their magnetic properties in the range 2–300 K.



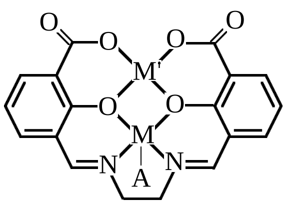
I



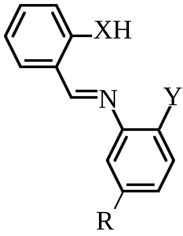
II

I, X, Y = NR, O, S; R = Alk, Ts; Ts = $SO_2C_6H_4Me$-*p*.

The use of polydentate Schiff bases for preparing antiferromagnetic and ferromagnetic complexes is well known. Kahn et al. [11–13] suggested structure **II** with two hydroxycarbonyl azomethine fragments. From this ligand, they prepared homo- and heterobinuclear coordination compounds **III** with various combinations of metals M and M'. At the similar ligand surrounding (including structures with an oxygen bridge), the complexes were either antiferromagnetic or ferromagnetic depending on particular combination of metal ions.



III



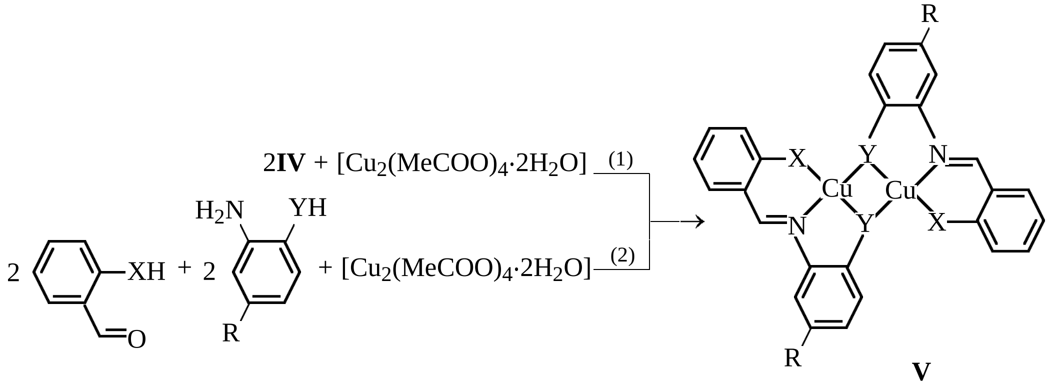
IV

III, M = Cu, Ni; M' = Cu, VO, Fe, Cr; A = none or Cl. **IV**, X = O, Y = NEt, R = NO_2 (**a**); X = NTs, Y = NEt, R = NO_2 (**b**); X = O, Y = NBu, R = NO_2 (**c**); X = NTs, Y = NBu, R = NO_2 (**d**); X = NTs, Y = O, R = H (**e**); X = O, Y = S, R = H (**f**).

The rational design of Kahn's model was accomplished in numerous later studies [14–20].

By the reaction of ligand system **IV** with copper acetate [pathway (1)] or by template synthesis [pathway (2)], we prepared homobinulcear copper chelates **V** with various ligand surroundings and varied Cu_2Y_2 core.

According to the analytical data, the composition of the coordination compounds corresponds to LM, which is consistent [21–31] with the formation of



V

V, X = O, Y = NC_2H_5, R = NO_2 (**a**); X = NTs, Y = NC_2H_5, R = NO_2 (**b**); X = O, Y = NC_4H_9, R = NO_2 (**c**); X = NTs, Y = NC_4H_9, R = NO_2 (**d**); X = NTs, Y = O, R = H (**e**); X = O, Y = S, R = H (**f**).

dimeric species of type **V**. Their structure is confirmed by the IR data, namely, by disappearance of the YH and XH stretching vibration bands (3300–3450 cm^{-1}) and decrease in the C=N stretching vibration frequencies (1600–1650 cm^{-1}) [23, 32]. The dimeric structure of chelates of type **V** was proved previously by single crystal X-ray diffractiomn for M = Co (X = NTs, Y = O [26]), Ni (X = Y = S [28], X = NTs, Y = O [25]), and Cu (X = NTs, Y = S [29]). Owing to the dimeric structure, copper chelates **V** give high-resolution ^1H NMR spectra.

Magnetochemical studies showed that most of the complexes under consideration are characterized by antiferromagnetic exchange coupling. The coupling is particularly strong in the compounds with the sulfur bridge, so that complex **V** with X = NTs, Y = S shows a diamagnetic behavior [29].

The experimental $\mu_{eff}(T)$ curves for complexes **V** are shown in Figs. 1–5. Assuming the dimeric structure of the complexes, we performed the theoretical simulation using the Bleaney–Bowers equation for the magnetic susceptibility of the dimer [4] [Eq. (1)] taking into account the exchange coupling between the dimers zJ' [Eq. (2)] and possible monomeric impurity p with the spin $S = 1/2$ [Eq. (3)].

$$\chi_{\text{Cu–Cu}} = \frac{N\beta^2 g^2}{3kT}\left(1 + \frac{1}{3}\exp\frac{-2J}{kT}\right)^{-1} + N\alpha, \qquad (1)$$

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

$$\chi = \chi'(1 - p) + \frac{N\beta^2 g^2 S(S+1)}{3k(T-\theta)}p, \qquad (3)$$

where N, $N\alpha$, k, b, J, g, and θ are, respectively, the Avogadro number, van Vleck paramagnetism, exchange coupling parameter, Lande factor, and Weiss constant.

The calculated optimal parameters are listed in the table.

Complexes **Va** and **Vc**–**Ve** are characterized by antiferromagnetic exchange. The quantity J (see table) depends on the fragments X and Y in the complex.

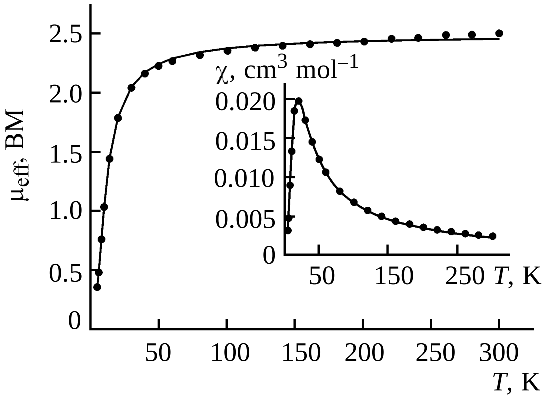


Fig. 1. Temperature dependence of the effective magnetic moment of **Va**. The theoretical dependence is shown by a solid line; the same for Figs. 2–4.

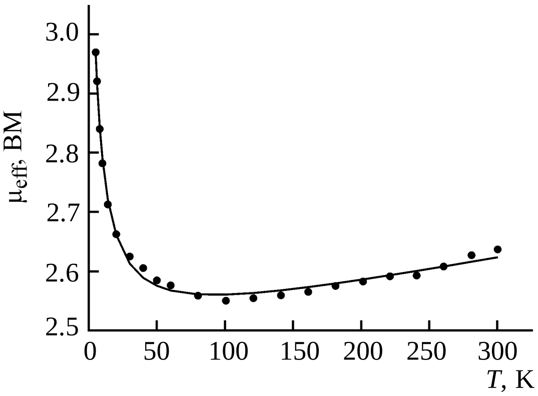


Fig. 2. Temperature dependence of the effective magnetic moment of **Vb**.

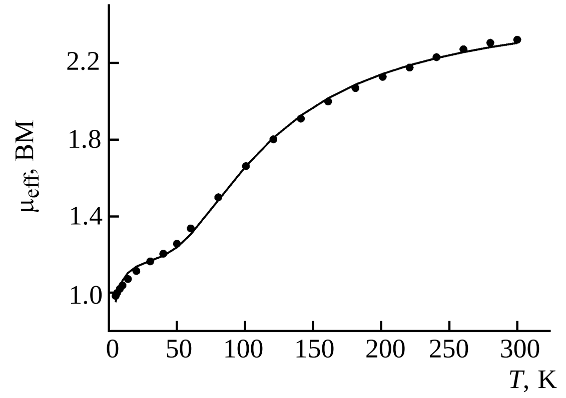


Fig. 3. Temperature dependence of the effective magnetic moment of **Vc**.

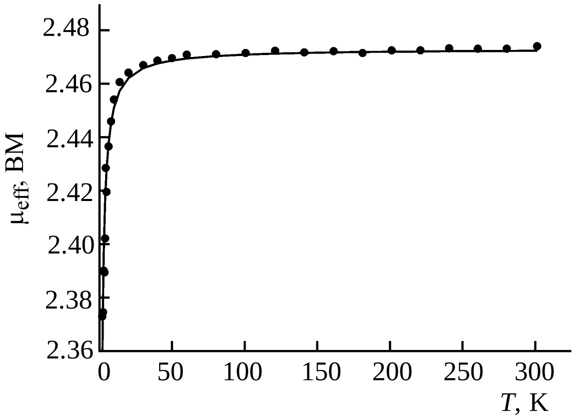


Fig. 4. Temperature dependence of the effective magnetic moment of **Vd**.

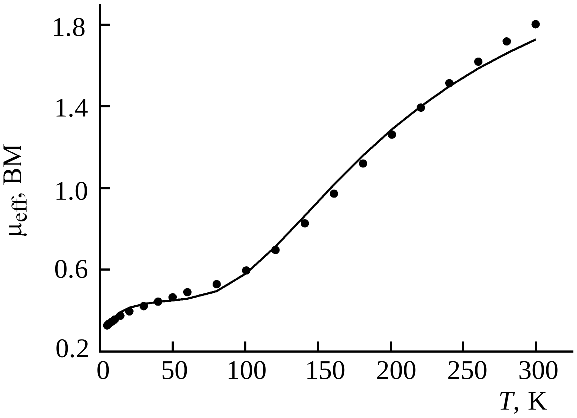


Fig. 5. Temperature dependence of the effective magnetic moment of **Ve**.

We found unexpectedly that compound **V** (X = NTs, Y = NEt) is characterized by positive ferromagnetic exchange (Fig. 2). It is difficult to explain this fact without comparative X-ray structural data for compounds **V** with X = NTs and Y = NEt and NBu.

EXPERIMENTAL

The IR spectra were recorded on a Nicolet Impact-400 spectrometer using mulls in mineral oil and KBr pellets. The ^{1}H NMR spectra were taken on a Varian Unity-300 device (300 MHz) in the mode of internal stabilization of the polar resonance ^{2}H line in $CDCl_3$. Magnetic measurements with **Va**–**Ve** were performed with a Quantum Design SQUID magnetometer in the range 2–300 K in a magnetic field of 5 kOe. In calculating the paramagnetic component of the magnetic susceptibility χ of complexes **Va**–**Ve**, we took into account the additive diamagnetic contribuiton of ions in accordance with the Pascal constants. The effective magnetic moment as a function of temperature was calculated by Eq. (4):

$$\mu_{eff}(T) = \left(\frac{3k}{N\beta^2}\chi T\right)^{1/2} \approx (8\chi T)^{1/2}. \qquad (4)$$

2,4-Dinitro-*N*-ethylaniline. To a suspension of 20.26 g of 2,4-dinitrochlorobenzene and 29.94 g of sodium acetate trihydrate ($CH_3COONa \cdot 3H_2O$) in 50 ml of ethanol, we added 8.97 g of ethylamine hydrochloride. The mixture was refluxed for 2 h and then cooled; the precipitate was filtered off and washed with water and ethanol. Yield 15.2 g (72%), mp 113–114°C (from ethanol), which agrees with published data [34].

2-Ethylamino-5-nitroaniline. A suspension of 21.1 g of 2,4-dinitro-*N*-ethylaniline in 40 ml of ethanol was added to a warm solution of 48.01 g of $Na_2S \cdot 9H_2O$ and 6.41 g of crystalline sulfur in 63 ml of water. The mixture was refluxed for 2 h, diluted with 30 ml of water, and cooled. The precipitate was filtered off and washed with warm water. Yield 12.5 g (69%), dark red crystals, mp 138–139°C (from ethanol), which agrees with published data [35].

2,4-Dinitro-*N*-butylaniline. To a suspension of 20.26 g of $CH_3COONa \cdot 3H_2O$ in 40 ml of ethanol, we

Magnetic properties of binuclear complexes

Comp. no.	g	J, cm^{-1}	zJ', cm^{-1}	p	$N\alpha$, cm^3 mol^{-1}
Va	2.05	–10	2.6		
Vb	2.02	4.03	0.27		0.00029
Vc[a]	2.13	–89		0.25	
Vd	2.02	–0.24			
Ve	2.15	–217		0.018	0.00026

[a] θ –3.3 K.

added 9.14 g of butylamine. The mixture was refluxed for 4 h and then cooled; the precipitate was filtered off and washed with water and ethanol. Yield 22.48 g (94%), yellow crystals, mp 89–90°C (from alcohol), which agrees with published data [36].

2-Butylamino-5-nitroaniline. A suspension of 23.92 g of 2,4-dinitro-*N*-butylaniline in 60 ml of alcohol was added to a warm solution of 48.01 g of $Na_2S\cdot 9H_2O$ and 6.41 g of crystalline sulfur in 60 ml of water. The mixture was refluxed for 210 min, diluted with 60 ml of water, and cooled. The precipitate was filtered off, washed with warm water, and dried. Yield 16.74 g (80%), brown crystals, mp 108–109°C (from ethanol). ^1H NMR spectrum, δ, ppm: 0.99 t (3H, CH_3, ^3J 7.3 Hz), 1.41–1.53 m (2H, CH_2), 1.64–1.74 m (2H, CH_2), 3.19–3.25 m (2H, CH_2), 3.31 br.s (2H, NH_2), 4.27 br.s (1H, NH), 6.54 d (1H, C_{Ar}–H, J_o 8.9 Hz), 7.63 d (1H, C_{Ar}–H, J_m 2.25 Hz), 7.84 d.d (1H, C_{Ar}–H, J_o 8.9, J_m 2.5 Hz). Found, %: C 57.31; H 7.33; N 20.15. $C_{10}H_{15}N_3O_2$. Calculated, %: C 57.40; H 7.23; N 20.08.

Compounds IVa and IVb *(general procedure).* A solution of 1.81 g of 2-ethylamino-5-nitroaniline in 100 ml of ethanol was added to a solution of 1.22 g of salicylaldehyde or 2.75 g 2-(tosylamino)benzaldehyde [37] in 30 ml of ethanol. The mixture was refluxed for 3 h. The precipitate that formed on cooling was filtered off and recrystallized from ethanol. **Compound IVa.** Yellow crystals, mp 194–195°C. IR spectrum, cm^{-1}: 3400 w (NH), 1615 s (C=N), 1275 m (Ph–O). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.34 t (3H, CH_3, ^3J 7.2 Hz), 3.31–3.40 m (2H, CH_2), 5.07 t (1H, NH, ^3J 4.9 Hz), 6.62–8.15 m (7H, C_{Ar}–H), 8.71 s (1H, CH=N), 12.31 s (1H, OH). Found, %: C 63.25; H 5.15; N 14.83. $C_{15}H_{15}N_3O_3$. Calculated, %: C 63.15; H 5.30; N 14.73. **Compound IVb.** Orange needles, mp 141–142°C. IR spectrum, cm^{-1}: 3446 w (NH), 1618 s (C=N), 1349 vs [$\nu_{as}(SO_2)$], 1168 vs [$\nu_s(SO_2)$]. ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.42 m (3H, CH_2–C*H*$_3$, ^3J 7.2 Hz), 2.38 s (3H, CH_3), 3.39–3.43 m (2H, C*H*$_2$–CH_3), 5.51 t (1H, NH–C_2H_5, ^3J 4.9 Hz), 6.67–8.17 m (11H, C_{Ar}–H), 8.68 s (1H, CH=N), 12.45 s (1H, NH). Found, %: C 60.16; H 5.16; N 12.88; S 7.42. $C_{22}H_{22}N_4O_4S$. Calculated, %: C 60.26; H 5.06; N 12.78; S 7.31.

Compounds IVe and IVf *(general procedure).* A solution of 0.55 g of *o*-aminophenol (Y = O) or 0.64 g of freshly distilled *o*-aminothiophenol (Y = S) in 10 ml of absolute toluene was added to a solution of 1.38 g of 2-(tosylamino)benzaldehyde [37] in 50 ml of absolute tolueme. The mixture was heated with a Dean–Stark trap until the calculated amount of water was completely removed. After the reaction completion, the solvent was distilled off on a rotary evaporator, and 30 ml of boiling methanol was added to the residue. The azomethine crystals that precipitated on cooling were filtered off, washed with two portions of methanol, and recrystallized from methanol. **Compound IVe.** Orange crystals, mp 142–143°C. IR spectrum, cm^{-1}: 3387 (OH), 1609 (C=N), 1332 [$\nu_{as}(SO_2)$], 1140 [$\nu_s(SO_2)$]. Found, %: C 65.56; H 5.02; N 8.19; S 8.90. $C_{20}H_{18}N_2O_3S$. Calculated, %: C 65.56; H 4.95; N 7.64; S 8.75. **Compound IVf.** Light yellow crystals, mp 210–211°C. IR spectrum, cm^{-1}: 1606 (C=N), 1360 [$\nu_{as}(SO_2)$], 1167 [$\nu_s(SO_2)$]. Found, %: C 63.04; H 5.04; N 7.05; S 16.50. $C_{20}H_{18}N_2O_2S_2$. Calculated, %: C 62.80; H 4.74; N 7.32; S 16.77.

Compounds Va and Vb *(general procedure).* A solution of 0.199 g of copper acetate monohydrate in 10 ml of ethanol was added to a solution of 0.283 g of azomethine **IVa** or 0.438 g of azomethine **IVb** in 50 ml of ethanol. The mixture was refluxed for 4 h. The crystals that formed on cooling were filtered off, washed with 3.5 ml of boiling ethanol, and vacuum-dried at 100°C. **Compound Va.** Dark brown crystals, mp 234–235°C (dec.). IR spectrum, cm^{-1}: 1604 s (C=N), 1327 w (Ph–O). Found, %: C 52.01; H 3.72; Cu 18.42; N 12.32. $C_{30}H_{26}CuN_6O_6$. Calculated, %: C 51.95; H 3.78; Cu 18.32; N 12.12. **Compound Vb.** Dark brown crystals, mp >260°C. IR spectrum, cm^{-1}: 1603 vs (C=N), 1283 vs [$\nu_{as}(SO_2)$], 1136 vs [$\nu_s(SO_2)$]. Found, %: C 52.75; H 4.13; Cu 12.92; N 11.32; S 6.51. $C_{44}H_{40}Cu_2N_8S_2$. Calculated, %: C 52.84; H 4.03; Cu 12.71; N 11.20; S 6.41.

Compounds Vc–**Vf** *(general procedure).* A solution of 0.21 g of 2-butylamino-5-nitroaniline (for **Vc**, **Vd**), 0.11 g of *o*-aminophenol (for **Ve**), or 0.13 g of *o*-aminothiophenol (for **Vf**) in 20 ml of ethanol was added to a solution of 0.122 g of salicylaldehyde (for **Vc**, **Vf**) or 0.275 g of 2-(tosylamino)benzaldehyde (for **Vd**, **Ve**) in 20 ml of ethanol. A solution of 0.2 g of copper acetate monohydrate in 10 ml of ethanol was also added. The mixture was refluxed for 4 h. The precipitates that formed on cooling were filtered off, washed with 3.5 ml of ethanol, and vacuum-dried at 100°C. **Compound Vc.** Dark brown powder, mp >250°C. IR spectrum, cm^{-1}: 1604 (C=N), 1326 m (Ph–O). Found, %: C 54.66; H 4.68; Cu 17.01; N 11.32. $C_{34}H_{34}CuN_6O_6$. Calculated, %: C 54.46; H 4.57; Cu 16.95; N 10.21. **Compound Vd.** Brown powder, mp 194–195°C. IR spectrum, cm^{-1}: 1559 s (C=N), 1292 vs [$\nu_{as}(SO_2)$], 1140 s [$\nu_s(SO_2)$]. Found, %: C 54.62; H 4.63; Cu 12.15; N 10.65; S 6.18. $C_{48}H_{48}CuN_8O_8S_2$. Calculated, %: C 54.58; H 4.59; Cu 12.03; N 10.61; S 6.07. **Compound Ve.** Dark green powder, mp >250°C (dec.). IR spectrum, cm^{-1}: 1593 (C=N), 1273 [$\nu_{as}(SO_2)$], 1120 [$\nu_s(SO_2)$].

Found, %: C 56.64; H 4.08; Cu 14.07; N 6.75; S 8.00. $C_{40}H_{32}Cu_2N_4O_6S_2$. Calculated, %: C 56.13; H 3.77; Cu 14.85; N 6.55; S 7.49. **Compound Vf.** Dark green crystals, mp >250°C (dec.). IR spectrum, cm^{-1}: 1586 (C=N), 1320 [$\nu_{as}(SO_2)$], 1127 [$\nu_s(SO_2)$]. ^1H NMR spectrum (DMSO-d_6), δ, ppm: 2.3 s (6H, $2CH_3$), 6.7–7.7 m (24H, H_{arom}), 8.0 s (2H, HC=N). Found, %: C 54.23; H 3.77; Cu 14.90; N 6.38; S 14.89. $C_{40}H_{32} \cdot Cu_2N_4O_4S_4$. Calculated, %: C 54.10; H 3.63; Cu 14.31; N 6.30; S 14.44.

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