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Synthesis, X-Ray Spectral, and Magnetochemical Study of Copper Complexes Based on Tridentate Azomethines of 3-Allylsalicylaldehyde

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Abstract—Tridentate azomethine ligands (H_2L) based on 3-allylsalicylaldehyde and *o*-aminophenol, *o*-aminothiophenol, or 2-ethylamino-5-nitroaniline were synthesized and identified by IR and ^{1}H NMR spectroscopies. The structure of complexes (**Ia–Ic**) was studied by magnetochemistry and EXAFS spectroscopy. Complexes **Ia** and **Ib** (X = O, S) are binuclear and exhibit antiferromagnetic exchange interaction: $2J = -36.5$ and -950 cm^{-1}, respectively. Complex **Ic** (X = NC_2H_5) is mononuclear ($\mu_{eff} = 1.75\ \mu_B$ at 275 K and does not change as temperature decreases).

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The synthesis of binuclear metal complexes based on tridentate Schiff bases is closely connected with the possibility of varying the fine structure of the azomethine ligand system, which affects the structure and physicochemical properties of the complexes [1–7]. These binuclear complexes play an important role in the development of modern magnetochemistry and can serve as promising materials for the design of molecular magnets [8–14].

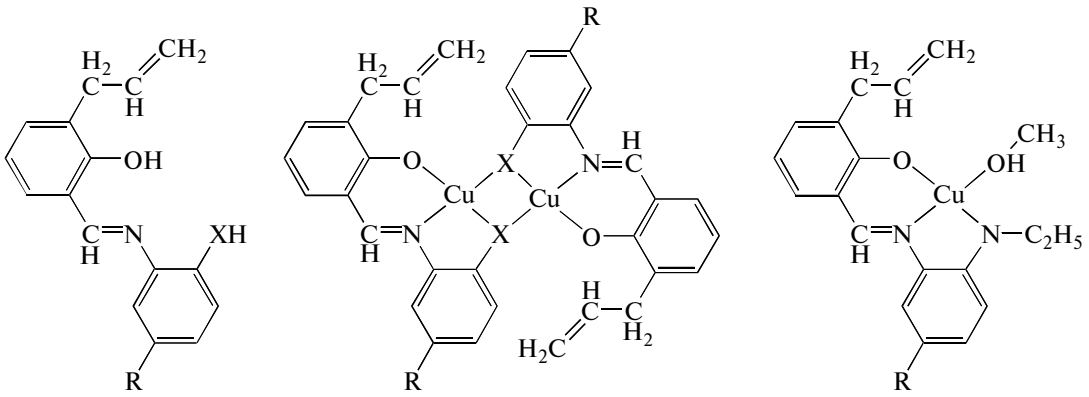
The key factors affecting the nature of magnetic exchange interactions in the dimeric complexes based on tridentate Schiff bases include the nature of the metal center, the type of bridging atoms and the nature of substituents in the amine and aldehyde moieties of Schiff bases.

In reviews [6, 7], it was shown that the substituents in the aldehyde and amine fragments of tridentate azomethines considerably affect the value and the nature of exchange interaction in the dimeric complexes. For example, the electron-withdrawing groups in position 5 of the aldehyde moiety enhance the antiferromagnetic interaction, while those in position 5 of the amine fragment reduce this interaction.

The replacement of the 2-hydroxy group in the aldehyde moiety by 2-tosylamino group not only increases the antiferromagnetic exchange in the dimeric molecule but, in some cases, alter the antiferromagnetic to ferromagnetic exchange type [15–23].

It was of interest to study the effect of the allyl group in position 3 of the aldehyde moiety on the strength and type of the exchange.

As a continuation of the synthesis and structure investigations of dimeric complexes based on tridentate azomethine ligands [15–23], we obtained new azomethine ligands (H_2L) based on 3-allylsalicylaldehyde and *o*-aminophenol, *o*-aminothiophenol, and 2-ethylamino-5-nitroaniline and their copper(II) complexes (**Ia–Ic**).



$(H_2L^{a-в})$: X = O, R = H (**a**); X = S, R = H (**b**); X = NC_2H_5, R = NO_2 (**c**)

(**Ia**, **Ib**)

(**Ic**)

EXPERIMENTAL

Commercially available (Alfa Aesar) 3-allylsalicylaldehyde, *o*-aminophenol, and *o*-aminothiophenol and 2-ethylamino-5-nitroaniline prepared by a reported procedure [17] were used.

Synthesis of azomethines. *o*-Aminophenol (1.03 g, 0.01 mol), or *o*-aminothiophenol (1.25 g, 0.01 mol), or 2-ethylamino-5-nitroaniline (1.81 g, 0.01 mol) in anhydrous toluene (10 mL) was added to a solution of 3-allylsalicylaldehyde (1.62 g, 0.01 mol) in anhydrous toluene (20 mL). The resulting mixture was heated with a Dean–Stark trap under argon until the calculated amount of water fully separated. After completion of the reaction, 2/3 of toluene was distilled off under vacuum, and boiling methanol (30 mL) was added. The precipitate was filtered off, washed with methanol (2 × 5 mL), and recrystallized from a methanol–toluene mixture (1 : 1).

3-Allylsalicylal-2'-hydroxyaniline H_2L^a (orange crystals, mp = 103–104°C). Yield 87%.

For $C_{16}H_{15}NO_2$			
calculated (%):	C, 75.87;	H, 5.97;	N, 5.53.
Found (%):	C, 75.90;	H, 6.01;	N, 5.58.

^{1}H NMR ($CDCl_3$; δ, ppm): 3.49 (2H, d, *J* = 6.6 Hz, $-\underline{CH_2}-CH=CH_2$), 5.09–5.16 (2H, m, $-CH_2-CH=\underline{CH_2}$), 5.82 (1H, s, OH), 6.01–6.10 (1H, m, $-CH_2-\underline{CH}=CH_2$), 6.92–7.26 (5H, m, $C_{Ar}-H$), 7.29–7.30 (2H, m, $C_{Ar}-H$), 8.69 (1H, s, CH=N), 12.55 (1H, s, OH). IR (ν, cm^{-1}): 3051 s ν(OH), 1626 s ν(C=N), 1282 w ν(Ph–O).

After evaporation of toluene, 3-allylsalicylal-2'-mercaptoaniline H_2L^b was used for the synthesis of **Ib** without isolation.

3-Allylsalicylal-2'-ethylamino-5'-nitroaniline H_2L^c (yellow crystals, mp = 174–175°C). Yield 55%.

For $C_{18}H_{19}N_3O_3$			
calculated (%):	C, 66.45;	H, 5.89;	N, 12.91.
Found (%):	C, 66.48;	H, 5.92;	N, 12.89.

^{1}H NMR ($CDCl_3$; δ, ppm): 1.35 (3H, t, *J* = 7.2 Hz, $-CH_2-\underline{CH_3}$), 3.36 (2H, q, *J* = 4.2 Hz, $-\underline{CH_2}-CH_3$), 3.49 (2H, d, *J* = 6.5 Hz, $-\underline{CH_2}-CH=CH_2$), 510–5.16 (3H, m, $-CH_2-CH=\underline{CH_2}$, NH), 6.01–6.09 (1H, m, $-CH_2-\underline{CH}=CH_2$), 6.63 (1H, d, *J* = 9.1 Hz, $C_{Ar}-H$), 6.97 (1H, t, *J* = 7.6 Hz, $C_{Ar}-H$), 7.26–7.36 (2H, m, $C_{Ar}-H$), 7.92 (1H, d, *J* = 2.5 Hz, $C_{Ar}-H$), 8.12 (1H, dd, *J* = 9.1 Hz, *J* = 2.4 Hz, $C_{Ar}-H$), 8.70 (1H, s, CH=N), 12.55 (1H, s, OH). IR (ν, cm^{-1}): 3402 s ν(NH), 3083 s ν(OH), 1610 s ν(C=N), 1293 w ν(Ph–O).

Synthesis of metal complexes. Acetate monohydrate in anhydrous methanol (10 mL) was added to a solution of 1 mmol of **Ia** (0.25 g), or **Ib** (0.27 g), or **Ic** (0.33 g) in anhydrous methanol (20 mL). The resulting mixture was refluxed on a water bath under argon for 2 h. The precipitated complexes were filtered off, washed with methanol (3 × 5 mL), and dried in a vacuum drying oven at 150°C.

Bis(3-allylsalicylal-2'-hydroxyanilinato)dicopper(II) (**Ia**, X = O, R = H). Dark green powder. mp > 260°C. Yield 80%.

For $C_{32}H_{26}N_2O_4Cu_2$				
calculated (%):	C, 61.04;	H, 4.16;	N, 4.45;	Cu, 20.19.
Found (%):	C, 61.15;	H, 4.05;	N, 4.32;	Cu, 20.21.

IR (ν, cm^{-1}): 1602 s ν(C=N), 1327 w ν(Ph–O).

Bis(3-allylsalicylal-2'-mercaptoanilinato)dicopper(II) (**Ib**, X = S, R = H). Dark brown powder. mp = 255–256°C. Yield 75%.

For $C_{32}H_{26}N_2O_2S_2Cu_2$

calculated (%): C, 58.07; H, 3.96; N, 4.23; Cu, 19.20.

Found (%): C, 58.17; H, 3.89; N, 4.32; Cu, 19.34.

IR (ν, cm^{-1}): 1601 vs ν(C=N), 1341 w (Ph–O).

[(Methanol)-3-allylsalicylal-2'-ethylamino-5-nitroanilinato]copper(II) (**Ic**, X = NC_2H_5, R = NO_2). Brown powder. mp = 255–256°C. Yield 56%.

For $C_{19}H_{21}N_3O_4Cu$

calculated (%): C, 54.47; H, 5.05; N, 10.03; Cu, 15.17.

Found (%): C, 54.52; H, 5.05; N, 10.15; Cu, 15.37.

IR (ν, cm^{-1}): 1602 vs ν(C=N), 1341 w ν(Ph–O).

IR spectra were recorded on a Varian 3100-FTIR Excalibur instrument for powders using the frustrated total internal reflection technique. ^1H NMR spectra were run on a Varian Unity-300 spectrometer (300 MHz) in the mode of internal stabilization of the ^2H polar resonance line in $CDCl_3$.

Cu *K*-edge X-ray absorption spectra were measured in the transmission mode on the EXAFS spectrometer of the station K1.3b "Structural material science" of the Kurchatov Synchrotron Center (Moscow). The energy of the electron beam used as the source of X-ray synchrotron radiation was 2.5 GeV at an average current of 60–80 mA. A Si(111) double crystal monochromator was utilized for energy selection at the Cu*K*-edge. The incident and transmitted X-ray radiation intensities were measured by air-filled ionization chambers connected to Keithley-6487 picoamperemeters.

The samples for recording the EXAFS spectra were thoroughly mixed with Apiezon and placed between thin polyester films. The sample thickness was so adjusted as to reduce the transmitted X-ray intensity by a factor of 2.5–3.0. After standard procedures for background subtraction, normalization to the *K*-edge step, and subtraction of atomic absorption μ_0 [24], the resulting EXAFS (χ)-spectra were Fourier transformed in the photoelectron wave vector *k* range from 2.5–3.5 to 12.5–15 $Å^{-1}$ with the weighting function k^3. The threshold ionization energy E_0 was chosen as the maximum of the *K*-edge first derivative and was subsequently varied during fitting. The exact values for the structure parameters of metal atom local environment were determined by non-linear fitting of the coordination sphere (CS) parameters. This was done by comparing the calculated EXAFS signal with the signal isolated from the full EXAFS spectrum by Fourier filtration of the Fourier transforms (FT). The non-linear fitting was done using the IFFEFIT-1.2.10 program package [25]. The photoelectron wave scattering phases and amplitudes needed to construct the theoretical spectrum were calculated using the FEFF7 program [26]. As model compounds, complexes of the same metals with similar local structures and with X-ray diffraction data available from the Cambridge Crystallographic Data Centre (CCDC) were used [27].

The goodness-of-fit function *Q*, which was minimized during determination of the structure parameters of the local environment, was calculated by the formula (1)

$$Q = \frac{\sum\left[k\chi_{exp}(k) - k\chi_{th}(k)\right]^2}{\sum\left[k\chi_{exp}(k)\right]^2} \times 100\%. \quad (1)$$

The magnetic properties of polycrystalline samples were studied on a Quantum Design MPMS-XL SQUID magnetometer in the temperature range of 2–300 K in a 5 kOe magnetic field. The calculated molar magnetic susceptibility was corrected for atom diamagnetism according to the Pascal additive method [11].

RESULTS AND DISCUSSION

The composition and structure of azomethines $H_2L^{a,\,в}$ were determined by elemental analysis, and ^1H NMR and IR spectroscopy.

The ^1H NMR spectrum of the ligand H_2L^a exhibits OH proton signals for the 3-allylsalicylaldehyde fragment at 12.55 ppm and for the *o*-aminophenol moiety at 5.82 ppm and CH=N signals at 8.69 ppm. The IR spectrum of H_2L^a shows a broadened stretching vibration band at 3051 cm^{-1} ν(OH) and stretching vibration bands at 1626 ν(C=N) and 1282 cm^{-1} ν(Ph–O).

The ^1H NMR spectrum of the ligand H_2L^b exhibits OH proton signals for the aldehyde moiety at 12.55 ppm and the CH=N group protons at 8.70 ppm. The amine (NH) and allyl (CH_2–CH=CH_2) protons are responsible for a multiplet at 5.10–5.16 ppm. The IR spectra of H_2L^b show ν(OH) and ν(NH) absorption bands at 3402–3083 cm^{-1}, a strong bands typical of the azomethine bond ν(C=N) at 1610 cm^{-1}, and a ν(Ph–O) band at 1293 cm^{-1}.

This spectral behavior in combination with the data of elemental analysis confirm the formation of imine structures for $H_2L^{a–c}$.

According to elemental analysis data, the composition of chelates **Ia** and **Ib** is $(ML)_2$. The changes in the IR spectra of these complexes compared to the spectra of ligands are typical for metal-chelates; in particular, the bands due to ν(OH) disappear, while the ν(C=N) frequency decreases by 10 cm^{-1} and the ν(Ph–O) frequency increases being manifested at 1327 (**Ia**) and 1341 cm^{-1} (**Ib**).

According to published data [15–23], complexes **Ia** and **Ib** may exist as dimers. A study of the temperature dependences of the magnetic susceptibility of **Ia** (Fig. 1) and **Ib** showed that their effective magnetic moments considerably decrease on cooling, that testify the antiferromagnetic exchange interaction between the copper(II) ions in these compounds.

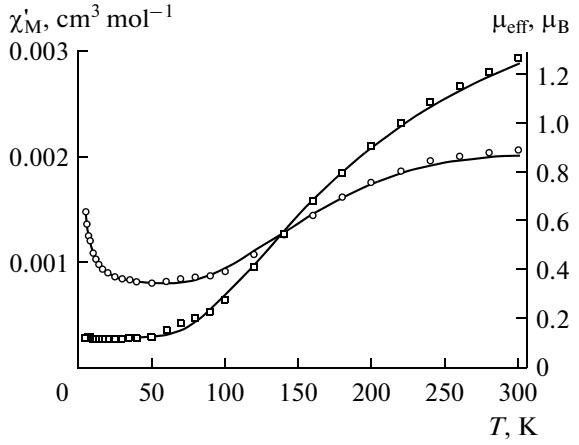


Fig. 1. Temperature dependence of (□) μ_{eff} per copper(II) atom and (○) χ'_M for complex **Ia** (the continuous line is the theoretical dependence).

The temperature dependence of the magnetic susceptibility of complexes is well interpreted within the framework of the Heisenberg–Dirac–Van Vleck isotropic model [11]. The exchange interaction parameters in complexes **Ia** and **Ib** were calculated by the Bleaney–Bowers equation [28].

The best agreement between the theory and experiment for complex **Ia** was observed for the following model parameters: $2J = -365$ cm^{-1}, $f = 0.0045$, $N_\alpha = 360 \times 10^{-6}$ cm^3/mol.

In complex **Ib**, the antiferromagnetic exchange interaction between the copper(II) ions is so strong that at temperatures below 200 K, the paramagnetism is caused by the presence of a minor paramagnetic impurity ($f \approx 0.05$). The difference between the magnetic properties of complexes **Ia** and **Ib** is caused by the difference in the nature of bridging atoms; the replacement of the bridging oxygen atoms by the sulfur atoms significantly increases the antiferromagnetic exchange in all known cases [6, 7, 16].

According to elemental analysis data, complex **Ic** has the composition CuL · CH_3OH. The effective magnetic moment of complex **Ic** does not depends on the temperature, being 1.75 μ_B at 2 K, which is practically equal to the purely spin value. These data may imply that complex **Ic** is mononuclear. In view of this conclusion, the model structure of complex **Ic** can be constructed considering two approximations: dimerization through the phenoxide oxygen atom is prevented by the *ortho* allyl substituent; dimerization through the nitrogen atom bonded to ethyl is unlikely.

In order to establish the local atomic structure of complexes **Ia–Ic**, copper *K*-edge X-ray absorption spectra (EXAFS) of these compounds were measured. Figure 2 shows the FT of EXAFS absorption for complexes **Ia–Ic**.

The FT for complexes **Ia–Ic** are substantially dissimilar. Whereas the FT of **Ia** and **Ic** exhibits one major peak ($r = 1.56$–1.59 Å) corresponding to the first CS, the FT of **Ib** shows two clearly resolved peaks ($r = 1.51$ and 1.92 Å) of approximately equal amplitudes. How-

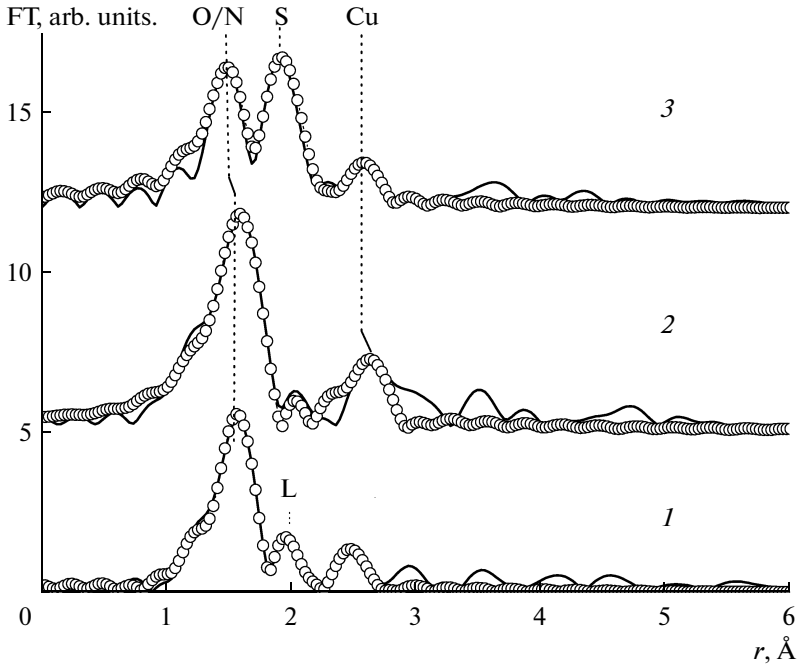


Fig. 2. FT for the best fit of complexes (*1*) **Ic**, (*2*) **Ia**, (*3*) **Ib**. (–) is experimental curve, (○) is theoretical FT.

ever, all FT have one feature in common, namely, a peak at r = 2.48–2.62 Å, which may reflect the Cu···Cu distance in the FT. By varying the lower and upper EXAFS integration parameters during Fourier transformation, it is possible to distinguish (against the CS containing light O and N atoms) CS with heavier Cu atoms due to different scattering amplitudes of photoelectrons by these atoms. Thus, it can be reliably stated that these peaks in the FT of complexes **Ia** and **Ib** correspond to the CS containing Cu atoms, indicating the dimeric structure of the molecules of these compounds. In the FT of complex **Ic**, the peak at r = 2.48 Å, conversely, corresponds to the CS consisting of light atoms and does not contain metal atoms. Note also the peak at r = 1.95 Å in the FT of **Ic**, which is absent in the other two FT. This peak was interpreted as being due to the solvent molecule coordinated to copper. Considering these FT features, the models of the dimeric complexes **Ia** and **Ib** were constructed where two copper ions are connected by two oxygen (**Ia**) or sulfur (**Ib**) bridges to each other and are bonded to two ligands (Cu–O and/or Cu–N bonds). Complex **Ic** is monomeric and has a coordinated solvent molecule.

By non-linear multisphere approximation of the calculated EXAFS to the experimental spectrum, quantitative CS parameters were found (table). For complexes **Ia** and **Ib**, the Cu···Cu distances are 3.02 ± 0.03 and 2.96 ± 0.03 Å, respectively. These distances are typical of dimeric compounds with similar ligands [7, 15, 16, 22, 23]. Statistical analysis of the corresponding Cu···Cu distances for more than 100 crystallographically characterized copper complex dimers from the CCDC also demonstrated that these distances for complexes **Ia** and **Ib** are close to the statistically mean values for complexes with sulfur (2.92 Å) and oxygen (3.0 Å) bridges (Fig. 3). The Cu···Cu distances in dimers with different bridges are different because the bond angle at a bridging sulfur atom is most often much smaller (<90°) than that at a bridging oxygen atom (>100°). This is caused by the fact that sulfur provides almost non-hybridized p orbitals for the overlap with metal atoms, whereas oxygen atoms tend to bind to metal atoms with pronounced sp hybridization.

The CS of complex **Ic** has a radius of 2.27 Å and contains atoms present in the coordinated solvent molecule. Note that for complex **Ib**, a satisfactory agreement of the Q factor was obtained only for the CS (C.N. 0.75) corresponding to the Cu···Cu distance at a constant typical value of the Debye–Waller factor. This may be indicative of either variability of Cu···Cu distances in this compound or a small monomer impurity.

Thus, by magnetochemical and X-ray spectral measurements, we found that, depending on the type of bridging X atom in the ligand molecule, copper binuclear complexes with strong antiferromagnetic

Structural data for the local atomic environment of the copper atom obtained by multisphere fitting of EXAFS data*

Compound	C.N.	R, Å	σ^2, Å^2	Atom	Q, %**
Ia	1	1.86	0.0030	N	9.9
	3	1.94	0.0030	O	
	1	3.02	0.0066	Cu	
Ib	1	1.90	0.0027	O/N	11.4
	1	1.96	0.0030	O/N	
	2	2.28	0.0062	S	
	0.75	2.96	0.0082	Cu	
Ic	2	1.95	0.0040	O/N	6.4
	2	1.99	0.0040	O/N	
	1	2.27	0.0040	L	

Notes: * R are interatomic distances, σ^2 is the Debye–Waller factor, Q is the goodness-of-fit function.
** The fitting was performed for R = 0.998–2.860 Å.
C.N. is the coordination number.

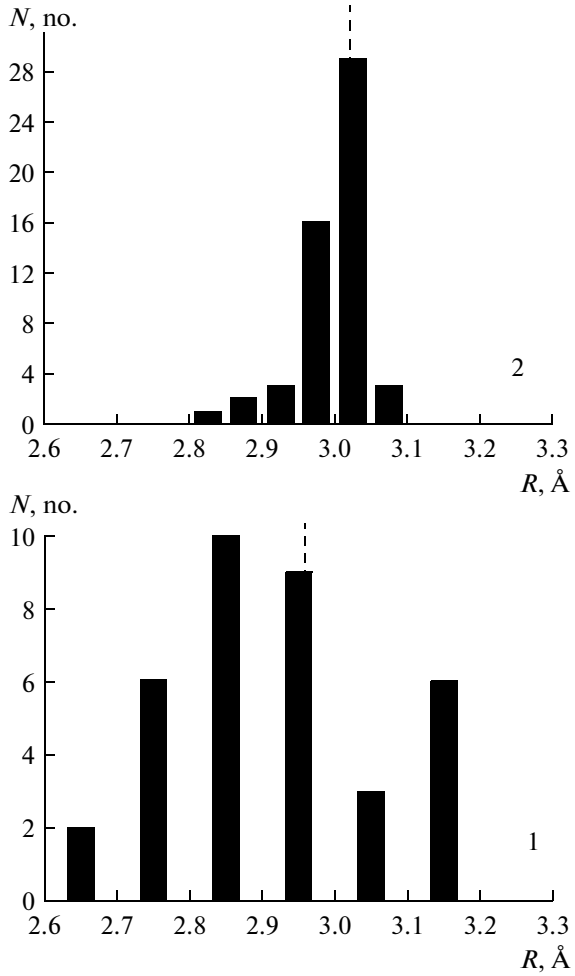


Fig. 3. Column diagram of the Cu···Cu distances in copper complex dimers with (1) S and (2) O bridges according to CCDC data. The dashed line shows the values for the Cu···Cu distances in **Ia** and **Ib**.

exchange interaction (X = O, S) or mononuclear complexes ($X = NC_2H_5$) are formed.

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