ISSN 1070-3284, Russian Journal of Coordination Chemistry, 2007, Vol. 33, No. 9, pp. 674–679. © Pleiades Publishing, Ltd., 2007.
Original Russian Text © V.T. Panushkin, T.E. Apenysheva, V.I. Sokol, V.S. Sergienko, K.S. Pushkareva, S.N. Bolotin, F.A. Kolokolov, E.V. Gromachevskaya, A.A. Borodavko, T.P. Kosulina, 2007, published in Koordinatsionnaya Khimiya, 2007, Vol. 33, No. 9, pp. 686–691.

The Copper(II) Acetate Complex with 2-(2-Hydroxyphenyl)-4,4-diphenyl-1,2-dihydro-4H-3,1-benzoxazine

V. T. Panushkin[a], **T. E. Apenysheva**[a], **V. I. Sokol**[b], **V. S. Sergienko**[b], **K. S. Pushkareva**[a], **S. N. Bolotin**[a], **F. A. Kolokolov**[a], **E. V. Gromachevskaya**[c], **A. A. Borodavko**[c], **and T. P. Kosulina**[c]

[a] *Kuban' State University, ul. Stavropol'skaya 129, Krasnodar 350040 Russia*

[b] *Kurnakov Institute of General and Inorganic Chemistry, Russian Academy of Sciences, Leninskii pr. 31, Moscow 119991 Russia*

[c] *Kuban' State Technological University, Krasnodar, Russia*

Received September 25, 2006

Abstract—The Cu(II) acetate complex with 2-(2-hydroxyphenyl)-4,4-diphenyl-1,2-dihydro-4H-3,1-benzoxazine (L) was synthesized for the first time and structurally studied by X-ray diffraction. The complex crystallizes as two crystallographically independent centrosymmetric binuclear molecules $[Cu_2(\mu\text{-}L)_2Ac_2]$ of similar structure, where L occurs as azomethin tautomeric form. The ligand performs tridentate chelate-bridging function. Each Cu atom exhibits extended tetragonal-pyramidal coordination by two O atoms and the N atom of one ligand L in the equatorial plane and by the O atom of the second ligand L in the axial position. The fourth equatorial position in the metal coordination polyhedron is occupied by the O atom of monodentate terminal acetate group.

DOI: 10.1134/S1070328407090096

1,2-Dihydro-4H-3,1-benzoxazines are original chemical systems, whose peculiar reactivity and structures are specified by annelation of 1,3-oxazine cycle with benzene nucleus. Since the above compounds have the hydroxyphenyl radical as a substituent in position 2, they are the potential tridentate ligands capable of complex formation with the transition metals. The formation of chelates can occur due to the elimination of the acid phenol proton with the formation of the ionic bond O–M and the donor-acceptor bonds M–O, M–N with the N and O atoms of benzoxazine ring.

The goal of this work was to synthesize the solid Cu(II) acetate complex with 2-(2-hydroxyphenyl-4,4-diphenyl-1,2-dihydro-4H-3,1-benzoxazine (**I**) and to study its composition and structure by X-ray diffraction.

EXPERIMENTAL

Synthesis. $Cu(CH_3COO)_2$ (0.0200 g) in 2.5 ml ethanol was added to compound L (0.0379 g) in 2.5 ml ethanol. The mixture was heated with a reflux condenser (50–60°C) until the dark blue precipitate of complex **I** was formed. After cooling, the precipitate was filtered, washed with cold ethanol, and dried in air. Single crystals were grown from the chloroform–alcohol mixture (1 : 1). The yield was 87%.

For $C_{56}H_{46}N_2O_8Cu_2$

anal. calcd. (%):	C, 69.69;	H, 4.53;	N, 2.84;	Cu, 12.98.
Found, (%):	C, 69.41;	H, 4.48;	N, 2.34;	Cu, 11.3.

X-ray diffraction analysis. The rhombohedral crystals of compound **I** of the dark blue color belong to the triclinic crystal system. The unit cell parameters: $a = 10.665(3)$, $b = 14.322(4)$, $c = 16.683(4)$ Å, $\alpha = 91.49(7)°$, $\beta = 98.55(6)°$, $\gamma = 107.63(8)°$, $V = 2394(1)$ Å^3, ρ(calcd.) = 1.317 g/cm^3, $\mu_{Mo} = 9.42$ cm^{-1}, $F(000) = 980$, $M = 949.96$, $Z = 2$, space group $P\bar{1}$.

The set of experimental data was obtained from a single crystal at room temperature on an Enraf-Nonius CAD-4 automated four-circle diffractometer (MoK_α radiation, graphite monochromator, ω-scan mode, $2\theta_{max} = 54°$). The total of 10811 reflections was recorded ($R_{int} = 0.035$, $-13 \le h \le 13$, $-18 \le k \le 18$, $0 \le l \le 21$).

The structure was solved by the direct method (SHELXS-97) [1] and refined by the full-matrix least-squares method (on F^2) for non-hydrogen atoms (SHELXL-97) [2]. The H(1) and H(1') atoms at O(2) and O(2'), respectively, were localized from the Fourier difference synthesis and refined in isotropic approximation. The positions of the remaining hydrogen atoms were calculated geometrically (C–H(Ph) 0.93, C–H(Me) 0.96 Å) and included in refinement with fixed positional and thermal parameters U_H. The latter are 0.1–0.3 Å^2 as large as the U_j parameters of the corresponding C atoms.

The final refinement parameters: $R_1 = 0.044$, $wR_2 = 0.108$ for 5875 reflections with $F_o \ge 4\sigma(F_o)$; $R_1 = 0.116$, $wR_2 = 0.135$ for all reflections; GOOF = 1.003. The maximum and minimum of residual electron density is

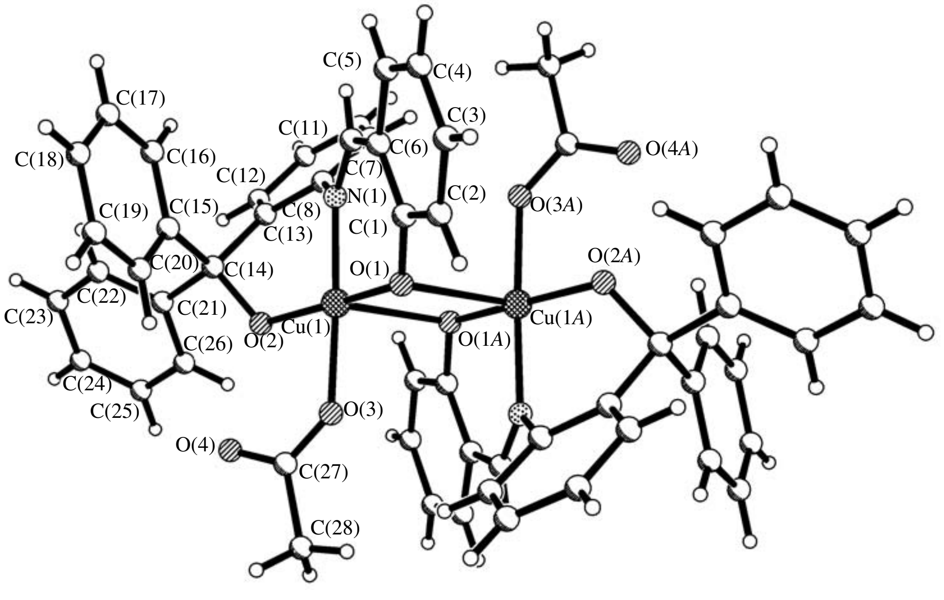


Fig. 1. The molecular structure of $[Cu_2L_2Ac_2]$.

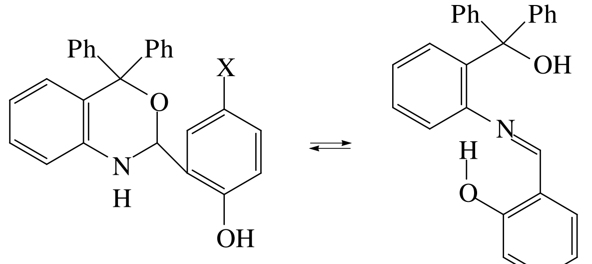
0.425 and –0.528 *e* Å^3; the coefficient of extinction is equal to 0.0000(3).

The coordinates and thermal parameters of atoms in structure **I** are given in Table 1; the bond lengths and bond angles are listed in Table 2.

RESULTS AND DISCUSSION

Dihydrobenzoxazines with no substituents in position 4 are capable of tautomeric transformation to give a linear azomethin form (the Schiff base) [3–5]. This tendency is the most pronounced in compounds, where the alternative unsaturated structure is stabilized by the intramolecular hydrogen bond [6, 7].

As follows from IR spectrum of a solid ligand L [5] recorded as KBr pellet, the absorption band at 3329 cm^{-1} is due to the stretching vibrations of the N–H bond. Thus, the molecule of a solid ligand occurs as a cyclic form **A**:



(A) (B)

According to the elemental analysis data, the reaction of complex formation gives, CuLAc compounds (Ac$^-$ is the acetate ion CH_3COO^-) independent of the initial ratio of the components. Thus, the reaction of complex formation is as follows:

$$Cu(Ac)_2 + HL \xrightarrow{T = 50°C} CuLAc + HAc.$$

The triclinic cell of compound **I** contains two centrosymmetric crystallographically independent binuclear molecules of the complex $[Cu_2(\mu\text{-}L)_2Ac_2]$ (**I** and **I'**). Since the geometrical parameters of these molecules are close, the further discussion will cover only one of them (**I**) and the averaged values will be given for the analogous bonds and angles. In the dimeric complex **I** (Fig. 1), the monodeprotonated benzoxazine ligand L occurs in azomethin tautomeric form. The H(1) atom lies at the O(2) atom of the phenylcarbinol fragment of the ligand L and participates in the formation of a strong chelate hydrogen bond between the L molecule and the O(4) atom of the acetate group. All the three donor centers of the ligand L (two O atoms and the N atom) are involved in the coordination with the Cu atom. The ligand L in complex **I** acts as a tridentate chelate bridge and closes two six-membered metal rings in a molecule of the complex. The acetate group in **I** is the monodentate terminal group. The coordination polyhedron of the Cu atom is an extended tetragonal pyramid (**TP**). The double oxygen bridge in binuclear molecules of the complex is formed by the O(1) and O(1*A*) atoms of the ligand phenol fragments, and in the TP of one Cu atom, the O(1) atom lies at the vertex of a base (Cu(1)–O(1) 1.905(3) ± 0.005 Å), whereas in the TP of the inverted Cu(1*A*) atom, in the axial position with considerably elongated bond (Cu(1*A*)–O(1) 2.405(3) ± 0.005 Å). The TP base contains also the O(2) and N(1) atoms of the phenylcarbinol and azomethin fragments of the L ligand and the acetate O(3) atom. The longest bond in the TP base is Cu–O$_{Ac}$ 1.972(3) ± 0.002 Å; the bonds Cu–N 1.955(3) ± 0.001 Å and Cu–O(2) 1.940(3) ± 0.009 Å are somewhat shorter.

Table 1. The atomic coordinates and thermal parameters U_{eq}, U_H in the crystal structure of $[Cu_2(\mu\text{-}L)_2Ac_2]$

Atom	*x*	*y*	*z*	U_{eq}, U_H, Å^2	Atom	*x*	*y*	*z*	U_{eq}, U_H, Å^2
Cu(1)	0.9824(1)	0.0820(1)	–0.0587(1)	0.0371(1)	Cu(1')	1.5385(1)	0.4122(1)	–0.5388(1)	0.0349(1)
O(1)	0.8541(2)	–0.0227(2)	–0.0199(2)	0.0463(6)	O(1')	1.5588(2)	0.5058(2)	–0.4126(1)	0.0416(6)
O(2)	1.0925(3)	0.1909(2)	–0.1098(2)	0.0445(7)	O(2')	1.6418(3)	0.3249(2)	–0.5090(2)	0.0392(6)
H(1)	1.079(5)	0.174(4)	–0.151(3)	0.08(2)	H(1')	1.694(6)	0.345(5)	–0.522(4)	0.12(3)
O(3)	0.9704(3)	–0.0061(2)	–0.1545(1)	0.0461(6)	O(3')	1.7020(2)	0.5092(2)	–0.5623(2)	0.0440(6)
O(4)	1.0570(3)	0.1010(2)	–0.2413(2)	0.0544(7)	O(4')	1.8334(3)	0.4160(2)	–0.5658(2)	0.0668(9)
N(1)	0.9900(3)	0.1731(2)	0.0325(2)	0.0427(7)	N(1')	1.3775(3)	0.3107(2)	–0.5205(2)	0.0353(6)
C(1)	0.7647(4)	–0.0120(3)	0.0239(2)	0.0497(9)	C(1')	1.3173(4)	0.4626(3)	–0.6254(2)	0.0422(8)
C(2)	0.6530(4)	–0.0923(4)	0.0273(3)	0.068(1)	C(2')	1.2709(4)	0.5235(3)	–0.6785(2)	0.055(1)
H(2*A*)	0.642	–0.151	–0.001	0.06	H(2*A*')	1.329	0.584	–0.686	0.06
C(3)	0.5587(5)	–0.0852(5)	0.0734(4)	0.087(2)	C(3')	1.1432(5)	0.4955(4)	–0.7185(3)	0.067(1)
H(3*A*)	0.483	–0.139	0.074	0.10	H(3*A*')	1.116	0.537	–0.754	0.08
C(4)	0.5740(6)	0.0000(5)	0.1187(4)	0.095(2)	C(4')	1.0537(5)	0.4062(4)	–0.7071(3)	0.072(1)
H(4*A*)	0.510	0.003	0.150	0.11	H(4*A*')	0.966	0.388	–0.735	0..08
C(5)	0.6828(5)	0.0798(4)	0.1178(3)	0.077(1)	C(5')	1.0938(4)	0.3450(3)	–0.6549(3)	0.058(1)
H(5*A*)	0.693	0.137	0.149	0.09	H(5*A*')	1.033	0.286	–0.646	0.06
C(6)	0.7810(4)	0.0754(3)	0.0696(2)	0.055(1)	C(6')	1.2262(3)	0.3704(3)	–0.6137(2)	0.0424(8)
C(7)	0.8967(4)	0.1593(3)	0.0762(2)	0.055(1)	C(7')	1.2598(4)	0.3044(3)	–0.5584(2)	0.0446(9)
H(7*A*)	0.906	0.209	0.116	0.06	H(7*A*')	1.190	0.251	–0.548	0.06
C(8)	1.1089(4)	0.2542(3)	0.0556(2)	0.0444(9)	C(8')	1.3892(3)	0.2444(2)	–0.4595(2)	0.0365(8)
C(9)	1.1709(5)	0.2708(3)	0.1358(2)	0.059(1)	C(9')	1.3031(4)	0.2279(3)	–0.4028(2)	0.050(1)
H(9*A*)	1.134	0.229	0.174	0.07	H(9*A*')	1.239	0.260	–0.405	0.05
C(10)	1.2861(5)	0.3469(4)	0.1598(3)	0.073(1)	C(10')	1.3118(4)	0.1640(3)	–0.3431(2)	0.056(1)
H(10*A*)	1.326	0.358	0.214	0.08	H(10*B*)	1.253	0.152	–0.306	0.06
C(11)	1.3420(5)	0.4072(3)	0.1033(3)	0.070(1)	C(11')	1.4074(4)	0.1182(3)	–0.3386(2)	0.052(1)
H(11*A*)	1.419	0.459	0.119	0.07	H(11*B*)	1.414	0.076	–0.298	0.07
C(12)	1.2829(4)	0.3900(3)	0.0223(2)	0.053(1)	C(12')	1.4941(4)	0.1351(2)	–0.3945(2)	0.0429(8)
H(12*A*)	1.322	0.430	–0.016	0.06	H(12*B*)	1.558	0.103	–0.391	0.05
C(13)	1.1658(4)	0.3137(3)	–0.0027(2)	0.0404(8)	C(13')	1.4885(3)	0.1981(2)	–0.4547(2)	0.0350(7)
C(14)	1.1006(3)	0.2921(2)	–0.0921(2)	0.0392(8)	C(14')	1.5847(3)	0.2200(2)	–0.5163(2)	0.0332(7)
C(15)	0.9594(4)	0.3015(3)	–0.1047(2)	0.0431(8)	C(15')	1.5102(3)	0.1831(2)	–0.6022(2)	0.0358(7)
C(16)	0.9258(4)	0.3649(3)	–0.0542(3)	0.060(1)	C(16')	1.4036(4)	0.0972(3)	–0.6160(2)	0.0458(9)
H(16*A*)	0.989	0.402	–0.011	0.07	H(16*B*)	1.376	0.062	–0.572	–0.05
C(17)	0.7987(5)	0.3738(4)	–0.0673(4)	0.084(2)	C(17')	1.3378(4)	0.0633(3)	–0.6935(3)	0.058(1)
H(17*A*)	0.777	0.417	–0.033	0.10	H(17*B*)	1.267	0.005	–0.701	0.06
C(18)	0.7054(5)	0.3203(4)	–0.1297(4)	0.085(2)	C(18')	1.3745(5)	0.1137(4)	–0.7589(3)	0.069(1)
H(18*A*)	0.620	0.326	–0.138	0.10	H(18*B*)	1.328	0.091	–0.811	0.08
C(19)	0.7375(5)	0.2581(4)	–0.1796(3)	0.084(2)	C(19')	1.4813(5)	0.1989(4)	–0.7461(3)	0.070(1)
H(19*A*)	0.674	0.221	–0.222	0.10	H(19*B*)	1.508	0.234	–0.790	0.06
C(20)	0.8653(4)	0.2485(3)	–0.1678(3)	0.061(1)	C(20')	1.5485(4)	0.2331(3)	–0.6693(2)	0.052(1)
H(20*A*)	0.887	0.206	–0.203	0.07	H(20*B*)	1.621	0.290	–0.662	0.06
C(21)	1.1866(4)	0.3565(3)	–0.1473(2)	0.0440(9)	C(21')	1.7019(3)	0.1794(2)	–0.4933(2)	0.0364(8)
C(22)	1.1748(4)	0.4467(3)	–0.1654(3)	0.067(1)	C(22')	1.7225(4)	0.1060(3)	–0.5410(2)	0.0458(9)
H(22*A*)	1.109	0.467	–0.146	0.08	H(22*B*)	1.664	0.080	–0.589	0.05
C(23)	1.2588(5)	0.5076(4)	–0.2116(3)	0.088(2)	C(23')	1.8288(4)	0.0712(3)	–0.5174(3)	0.060(1)
H(23*A*)	1.251	0.569	–0.222	0.11	H(23*B*)	1.841	0.022	–0.550	0.07
C(24)	1.3541(5)	0.4767(5)	–0.2413(3)	0.084(2)	C(24')	1.9153(4)	0.1076(3)	–0.4466(3)	0.060(1)
H(24*A*)	1.410	0.517	–0.273	0.08	H(24*B*)	1.987	0.083	–0.431	0.06
C(25)	1.3670(5)	0.3881(4)	–0.2246(3)	0.073(1)	C(25')	1.8961(4)	0.1807(3)	–0.3988(3)	0.053(1)
H(25*A*)	1.432	0.367	–0.245	0.08	H(25*B*)	1.954	0.206	–0.350	0.05
C(26)	1.2844(4)	0.3267(3)	–0.1770(3)	0.059(1)	C(26')	1.7915(4)	0.2160(3)	–0.4222(2)	0.0458(9)
H(26*A*)	1.295	0.266	–0.165	0.07	H(26*B*)	1.780	0.266	–0.390	0.05
C(27)	1.0116(4)	0.0159(3)	–0.2209(2)	0.0422(8)	C(27')	1.8117(4)	0.4973(3)	–0.5700(2)	0.0456(9)
C(28)	1.0049(5)	–0.0682(3)	–0.2796(2)	0.065(1)	C(28')	1.9209(4)	0.5843(3)	–0.5868(3)	0.061(1)
H(28*A*)	0.969	–0.129	–0.257	0.10	H(28*D*)	1.890	0.641	–0.589	0.09
H(28*B*)	1.092	–0.062	–0.290	0.10	H(28*E*)	1.946	0.572	–0.638	0.09
H(28*C*)	0.949	–0.066	–0.330	0.10	H(28*F*)	1.996	0.596	–0.544	0.09

Table 2. The bond lengths (*d*) and bond angles (ω) in [Cu$_2$(μ-L)$_2$Ac$_2$]

Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
Cu(1)–O(1)	1.900(2)	O(3)–C(27)	1.265(4)	Cu(1')–O(1*A*')	2.410(2)
Cu(1)–O(2)	1.949(3)	O(4)–C(27)	1.244(4)	O(1')–C(1')	1.314(5)
Cu(1)–O(3)	1.974(2)	N(1)–C(7)	1.291(5)	O(2')–H(1')	0.62(7)
Cu(1)–N(1)	1.955(3)	N(1)–C(8)	1.430(5)	O(2')–C(14')	1.433(4)
Cu(1)–O(1*A*)	2.400(2)	Cu(1')–O(1')	1.910(2)	O(3')–C(27')	1.257(5)
O(1)–C(1)	1.324(5)	Cu(1')–O(2')	1.931(3)	O(4')–C(27')	1.256(5)
O(2)–H(1)	0.69(5)	Cu(1')–O(3')	1.971(2)	N(1')–C(7')	1.294(5)
O(2)–C(14)	1.446(4)	Cu(1')–N(1')	1.955(3)	N(1')–C(8')	1.428(4)
Angle	ω, deg	Angle	ω, deg	Angle	ω, deg
O(1)Cu(1)O(2)	171.7(1)	N(1)C(7)C(6)	126.3(4)	O(1*A*')Cu(1')N(1')	93.3(6)
O(1)Cu(1)O(3)	88.5(1)	N(1)C(8)C(9)	119.5(4)	Cu(1')O(3')C(27')	128.9(2)
O(1)Cu(1)N(1)	92.6(1)	N(1)C(8)C(13)	120.5(3)	Cu(1')N(1')C(7')	122.7(3)
O(1)Cu(1)O(1*A*)	85.7(1)	O(2)C(14)C(21)	108.9(3)	Cu(1')N(1')C(8')	118.6(2)
O(2)Cu(1)O(3)	89.2(1)	O(3)C(27)O(4)	125.1(4)	C(7')N(1')C(8')	118.5(3)
O(2)Cu(1)O(1*A*)	102.3(1)	O(3)C(27)C(28)	117.0(3)	O(1')C(1')C(2')	118.8(3)
O(2)Cu(1)N(1)	89.3(1)	O(4)C(27)C(28)	117.9(3)	O(1')C(1')C(6')	123.2(3)
O(1A)Cu(1)O(3)	92.0(4)	O(1')Cu(1')O(2')	169.8(1)	N(1')C(7')C(6')	126.8(3)
O(1A)Cu(1)N(1)	90.8(4)	O(1')Cu(1')O(3')	87.8(1)	N(1')C(8')C(9')	119.2(3)
Cu(1)O(1)C(1)	125.0(2)	O(1')Cu(1')N(1')	93.3(1)	N(1')C(8')C(13')	121.0(3)
Cu(1)O(2)C(14)	123.0(2)	O(2')Cu(1')O(3')	88.7(1)	O(2')C(14')C(13')	104.8(3)
Cu(1)O(3)C(27)	128.6(2)	O(2')Cu(1')N(1')	89.7(1)	O(2')C(14')C(21')	105.8(3)
Cu(1)N(1)C(7)	122.8(3)	O(3')Cu(1')N(1')	176.6(1)	O(2')C(14')C(15')	111.2(3)
Cu(1)N(1)C(8)	118.3(2)	O(1*A*')Cu(1')O(1')	86.2(2)	O(3')C(27')O(4')	123.6(4)
O(1)C(1)C(2)	118.7(4)	O(1*A*')Cu(1')O(2')	169.8(6)	O(3')C(27')C(28')	118.0(3)
O(1)C(1)C(6)	122.6(4)	O(1*A*')Cu(1')O(3')	87.7(2)	O(4')C(27')C(28')	118.4(3)
C(7)N(1)C(8)	118.7(3)				

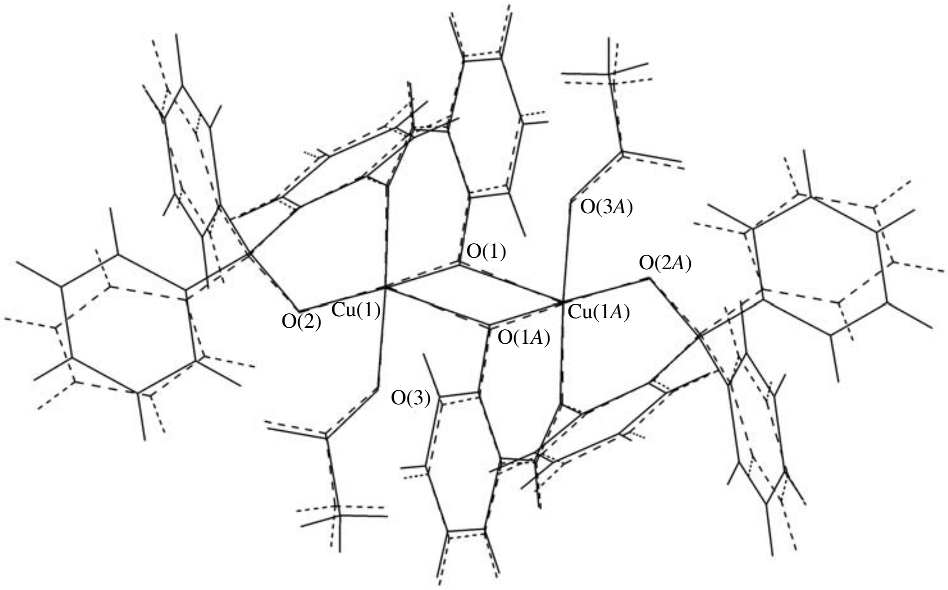


Fig. 2. Superposition of two independent molecules L (solid line) and L' (dotted line) provided that eight atoms coincide (Cu(1), Cu(1*A*), O(1), O(1*A*), O(2), O(2*A*), O(3), and O(3*A*)).

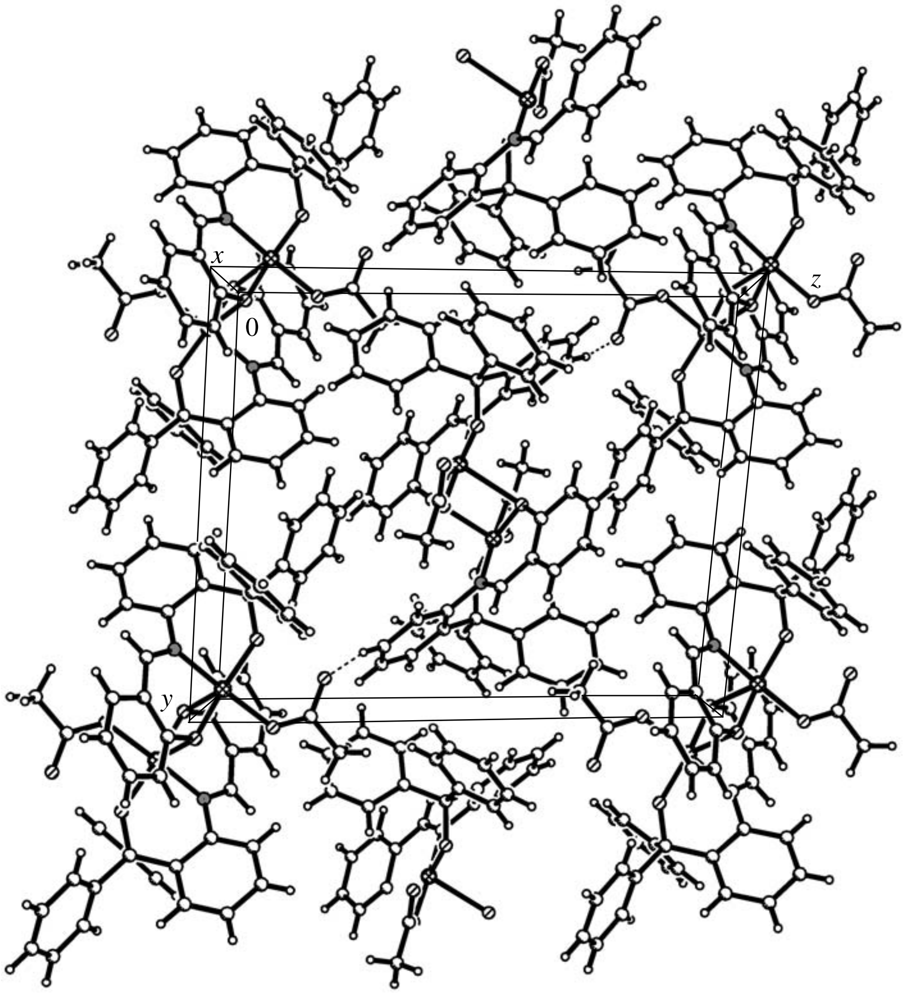


Fig. 3. The packing of the complex molecules in crystal **I** (projection onto the plane *yz*).

The greatest deviations from the ideal values of 180° and 90° are observed for the TP angles O(1*A*')Cu(2)O(2') 169.7° and O(2)Cu(1)O(1*A*) 103.3°. The Cu···Cu distance in the dimers is equal to 3.170 ± 0.003 Å.

The geometrical parameters of the chelate O(2)–H(1)···O(4) in the H-heterocycle of the complex are: O(2)–H 0.69(5), O(4)···H 1.78(5), O(2)···O(4) 2.430(4) Å, the angle O(2)H(1)O(4) is 168(3)°. The above H-cycle, like six-membered chelate metal cycles conjugated with the phenyl rings in complex **I**, has pronounced nonplanar structure. The H-cycle has the asymmetric boat conformation: the O(2) and C(27) atoms deviate from the mean plane Cu(1)O(3)O(4)H(1) (Δ = 0.04 Å) by 0.223 and 0.086 Å, respectively. The chelate cycle conjugated with the triphenylcarbinol group also has the asymmetric boat conformation: the N(1) and C(14) atoms extend from the Cu(1)O(2)C(8)C(13) plane (Δ = 0.03 Å) by 0.505 and 0.692 Å, respectively. The second cycle conjugated with the phenol ring has the sofa conformation: the Cu atom deviates by 0.501 Å from the O(1)N(1)C(1)C(6)C(7) plane (Δ = 0.03 Å).

As was noted above, the geometrical parameters of two independent ligands L and L' in the complexes **I** and **I'** are very close, particularly, the lengths of the analogous bonds in the ligands. The lengths of the bonds O–C(sp^3) in the triphenylcarbinol fragment O(2)–C(14) (1.446(4) ± 0.007 Å), O–C(sp^2) in the phenyl fragment O(1)-C(11) (1.311(5) ± 0.005 Å) and O=C(sp^2) in the acetate group O(3)–C(27) (1.261(5) ± 0.004 Å) and O(4)–C(27) (1.250(4) ± 0.006 Å) are close to the standard values [8]. The N(1)–C(7) bond 1.292(5) ± 0.002 Å in the ligand L is multiple.

The main difference between two independent fragments CuL and Cu'L' in structure **I** (Fig. 2) appears in different relative direction of the phenyl rings and acetate groups. The analogous angles between the planes of the phenyl rings in the triphenylcarbinol fragment in L and L' slightly differ and are equal to 76.3°, 89.9° and 97.9° in L and 81.7°, 86.7°, and 112.7° in L'.

The structural units of crystal **I**, i.e., the binuclear molecules of the complexes, are located at two independent inversion centers (0, 0, 0 and 0.5, 0.5, 0.5) (Fig. 3). They show different orientation relative to the inversion centers, which is most likely due to the optimum crystal packing requirements. The shortened contacts are observed between the complex molecules in the crystal. The shortest of them are O(3)···C(7) 3.163, O(4)···C(10') 3.323, and O(3')···C(24) 3.372 Å.

ACKNOWLEDGMENTS

This work was supported by the Russian Foundation for Basic Research (project no. 06-03-32881-a).

REFERENCES

1. Sheldrick, G.M., *SHELXS-97. Program for the Solution of Crystal Structures*, Göttingen (Germany): Univ. of Göttingen, 1997.
2. Sheldrick, G.M., *SHELXL-97. Program for the Refinement of Crystal Structures*, Göttingen (Germany): Univ. of Göttingen, 1997.
3. Gromachevskaya, E.V., Kosulina, T.P., and Kul'nevich, V.G., *Khim. Geterotsikl. Soedin.*, 1985, no. 12, p. 1682.
4. Kliege, W., Metge, J., and Retting, S.J., *Can. J. Chem.*, 1988, vol. 76, no. 4, p. 389.
5. Neuvonen, K., Pohtala, R., and Pihlaja, K., *Magn. Res. Chem.*, 1989, vol. 27, no. 8, p. 725.
6. Saeed, A.A.H. and Ebraheem, E.K., *Can. J. Spectrosc.*, 1983, no. 6, p. 169.
7. Gromachevskaya, E.V., Kosulina, T.P., Kul'nevich, V.G., et al., *Khim. Geterotsikl. Soedin.*, 1990, no. 1, p. 101.
8. Allen, F.H., Kennard, O., Watson, D., et al., *J. Chem. Soc., Perkin Trans. 2*, 1987, p. S1.