Cl3	0.8503 (2)	0.3503 (2)	0.7081 (3)	0.0801 (12)
N1	0.2897 (6)	0.7897 (6)	0.3923 (8)	0.045 (2)
C111	0.3802 (8)	0.7492 (9)	0.3248 (9)	0.067 (3)
C112	0.4323 (10)	0.6571 (10)	0.3765 (11)	0.092 (4)
C121	0.3227 (9)	0.8227 (9)	0.5087 (14)	0.076 (4)
C122	0.4039 (10)	0.9039 (10)	0.5107 (16)	0.089 (5)
C131	0.2095 (10)	0.7095 (10)	0.4090 (16)	0.087 (5)
C132	0.1659 (11)	0.6659 (11)	0.3019 (15)	0.090 (5)
N2	0	0	0	0.064 (5)
C2	0.0321 (10)	0.0869 (10)	−0.0709 (11)	0.081 (3)
N3	0	1/2	0.0054 (14)	0.056 (5)
C31†	−0.023 (5)	0.607 (3)	−0.031 (5)	0.105 (10)
C32†	−0.100 (3)	0.443 (4)	0.024 (4)	0.105 (10)
C33†	0.051 (4)	0.518 (4)	0.114 (3)	0.105 (10)
C34‡	0.060 (2)	0.441 (2)	−0.076 (4)	0.105 (10)

† Site occupancy = 0.25. ‡ Site occupancy = 0.50.

Table 2. *Selected geometric parameters (Å, °)*

Cu—Cl1	2.243 (3)	C121—C122	1.50 (2)
Cu—Cl2	2.242 (5)	C131—C132	1.51 (3)
Cu—Cl3	2.264 (5)	N2—C2	1.48 (1)
N1—C111	1.52 (1)	N3—C31	1.49 (4)
N1—C121	1.51 (2)	N3—C32	1.52 (4)
N1—C131	1.50 (2)	N3—C33	1.47 (3)
C111—C112	1.51 (2)	N3—C34	1.47 (3)
Cl1—Cu—Cl1^{i}	116.3 (2)	C132—C131—N1	115 (1)
Cl1—Cu—Cl2	105.6 (1)	C2—N2—C2^{iii}	110.4 (10)
Cl1—Cu—Cl3	105.5 (1)	C2—N2—C2^{iv}	109.0 (5)
Cl2—Cu—Cl3	118.9 (2)	C31—N3—C32	109 (4)
C111—N1—C111^{ii}	105.3 (10)	C31—N3—C33	101 (3)
C111—N1—C121	111.0 (7)	C31—N3—C34	114 (3)
C111—N1—C131	111.7 (7)	C32—N3—C33	110 (3)
C121—N1—C131	106.2 (11)	C32—N3—C34	107 (2)
C112—C111—N1	114.4 (9)	C33—N3—C34	115 (3)
C122—C121—N1	115 (1)		

Symmetry codes: (i) $\frac{1}{2}+y, x-\frac{1}{2}, z$; (ii) $y-\frac{1}{2}, \frac{1}{2}+x, z$; (iii) $-x, -y, z$; (iv) $-y, x, -z$.

The title structure was solved by the Patterson method, from subsequent difference-Fourier calculations and refined by full-matrix least-squares techniques with isotropic displacement parameters for non-H atoms, except Cu and Cl atoms which were refined anisotropically. The bond lengths of the disordered tetramethylammonium group were constrained [N—C = 1.45 (5) Å]. The H atoms were idealized with the standard methods in *SHELXL*93 (Sheldrick, 1993) and included in the least-squares process as fixed contributors; their isotropic displacement parameters were set at $1.5U_{iso}$ of the corresponding C atoms.

Data collection: Enraf–Nonius CAD-3 software. Cell refinement: Enraf–Nonius CAD-3 software. Data reduction: local program. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SK1018). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis[4-(5-chloro-2-hydroxyphenylimino-*N*,*O*:*O*)-2-penten-2-olato(2–)-*O*]dicopper(II)

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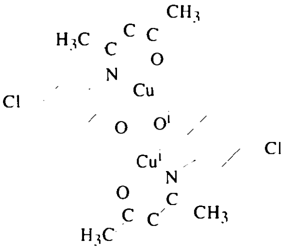
Abstract

In the title complex, $[Cu_2(C_{11}H_{10}ClNO_2)_2]$, the Cu atoms have two phenolic oxygen bridges resulting in centrosymmetric Cu dimers. The coordination around copper is distorted square planar, with an average Cu—O distance of 1.921 (5) and a Cu—N distance of 1.910 (5) Å. Two additional O atoms belonging to adjacent dimers at distances of 2.986 (6) and 3.139 (6) Å complete the distorted octahedron about copper.

Comment

Doubly oxygen-bridged dimeric copper(II) complexes with subnormal magnetic moments are known to exhibit super-exchange interaction (Kato, Jonassen & Fanning, 1964; Butcher & Sinn, 1976; Kato & Muto, 1988). The magnetic interactions depend on the immediate environment of the bridge, as well as on the ligand arrangement about the Cu atom. The structure determination of the title compound, (I), was undertaken in order to permit a comparison of its bridging environment with those of similar dimeric systems. The magnetic properties are currently under investigation.



(I)

The -Cu—O—Cu—O- bridging group is planar, as one would expect due to the centre of inversion at its centre (Fig. 1). The Cu—O bridging bond lengths [1.941 (5) and 1.950 (4) Å] are within the range of corresponding values observed in related Cu^{II} complexes (Butcher & Sinn, 1976; Kato & Muto, 1988). The bridging angles O1—Cu1—O1^i and Cu1—O1—Cu1^i are 79.4 (2) and 100.6 (2)°, respectively [symmetry code: (i) $1-x$, $-y$, $2-z$]. The Cu atoms have a distorted square-planar environment, with bond angles in the range 79.4 (2)–100.0 (2)°. The average Cu—O distance in the coordination square is 1.921 (5) Å, while the Cu—N distance has a value of 1.910 (5) Å. The -Cu—O—Cu—O- bridging plane and the coordination plane around copper make a dihedral angle of 5 (1)° with respect to one another. There are reports in the literature that this dihedral angle, as well as the Cu—O—Cu bridging angle, influences the magnetic properties of the systems. The Cu···Cu distance [2.994 (2) Å] in the bridging plane is rather long for a direct interaction. The dimer molecule as a whole is fairly planar, with a maximum dihedral angle of 29.3 (3)° between the planes through O1—C1—C2—C3—C4—C5—C6—N1 and N1—C7—C8—C9—O2. As a result, the molecules are stacked parallel to one another so that the closest distance between a Cu atom and the O atoms of neighbouring dimers above and below are 2.986 (6) [Cu1—O1^{ii}] and 3.139 (6) Å [Cu1—O2^{iv}], respectively (symmetry codes as in Table 2). With these two weak intermolecular interactions, the coordination around copper could be considered sixfold, which is a distorted octahedron. The Cl-substituted phenol has usual bond lengths and angles.

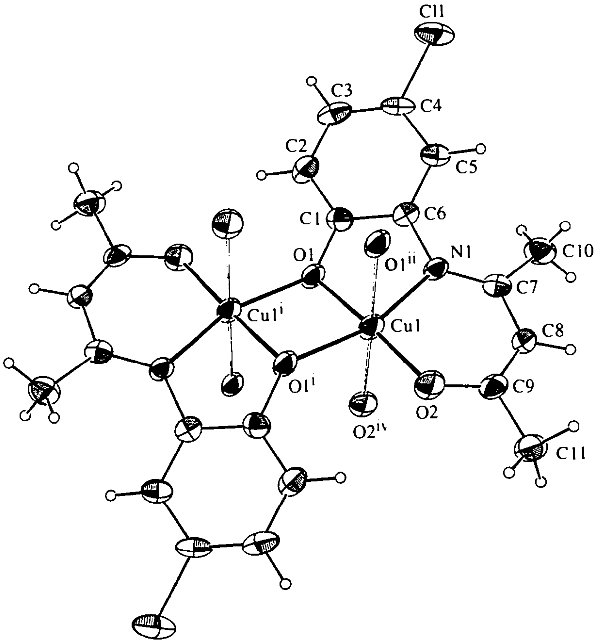


Fig. 1. The molecular structure of [Cu$_2$(C$_{11}$H$_{10}$ClNO$_2$)$_2$] with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small circles of arbitrary radii.

Experimental

A solution of Cu(CH$_3$COO)$_2$.H$_2$O (0.199 g, 1 mmol) in 30 ml methanol was added to a solution of *N*-(acetylacetylidine)-5-chloro-2-hydroxyaniline (0.225 g, 1 mmol) in 50 ml hot CH$_3$CN and the resulting mixture set aside for 1 h. The dark-green precipitate which formed was filtered, 0.2 g mixed with 60 ml DMF and the mixture heated to boiling. Crystals were obtained after allowing the solution to stand for 36 h at room temperature.

Crystal data

[Cu$_2$(C$_{11}$H$_{10}$ClNO$_2$)$_2$]
$M_r = 574.40$
Monoclinic
$P2_1/n$
$a = 12.175\,(2)$ Å
$b = 3.845\,(2)$ Å
$c = 22.715\,(1)$ Å
$\beta = 92.37\,(4)^\circ$
$V = 1062.4\,(6)$ Å^3
$Z = 2$
$D_x = 1.792$ Mg m^{-3}
D_m not measured

Mo *Kα* radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8.37–18.24^\circ$
$\mu = 2.29$ mm^{-1}
$T = 295$ K
Needle
0.45 × 0.15 × 0.10 mm
Dark blue

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (*MolEN*; Fair, 1990)
$T_{min} = 0.65$, $T_{max} = 0.80$
2543 measured reflections
2423 independent reflections

1161 observed reflections [$I > 2\sigma(I)$]
$R_{int} = 0.028$
$\theta_{max} = 26.32^\circ$
$h = -15 \rightarrow 15$
$k = 0 \rightarrow 4$
$l = -28 \rightarrow 0$
3 standard reflections
frequency: 120 min
intensity decay: 1.92%

Refinement

Refinement on *F*
$R = 0.049$
$wR = 0.052$
$S = 1.35$
1161 reflections
145 parameters
H atoms riding
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\max} = 0.00029$
$\Delta\rho_{\max} = 0.530$ e Å^{-3}
$\Delta\rho_{\min} = -0.383$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}
Cu1	0.47691 (7)	0.1828 (3)	0.94262 (4)	2.64 (2)
Cl1	1.0022 (2)	0.1002 (7)	0.8715 (1)	4.15 (5)
O1	0.5810 (3)	−0.1429 (15)	0.9789 (2)	2.8 (1)
O2	0.3691 (4)	0.4680 (15)	0.9065 (2)	3.3 (1)
N1	0.5757 (4)	0.1864 (16)	0.8795 (2)	2.0 (1)
C1	0.6807 (5)	−0.109 (2)	0.9543 (3)	2.2 (2)
C2	0.7773 (5)	−0.226 (2)	0.9813 (3)	2.6 (2)
C3	0.8753 (5)	−0.174 (2)	0.9550 (3)	2.9 (2)
C4	0.8771 (5)	0.003 (2)	0.9028 (3)	2.4 (2)
C5	0.7809 (5)	0.122 (2)	0.8737 (3)	2.4 (2)
C6	0.6805 (5)	0.0571 (18)	0.8995 (3)	1.8 (1)
C7	0.5428 (5)	0.2515 (18)	0.8245 (3)	1.9 (1)
C8	0.4409 (5)	0.4158 (19)	0.8118 (3)	2.2 (2)
C9	0.3632 (5)	0.512 (2)	0.8497 (3)	2.3 (2)
C10	0.6091 (5)	0.154 (2)	0.7731 (3)	3.1 (2)
C11	0.2577 (5)	0.684 (2)	0.8290 (3)	3.1 (2)

Table 2. *Selected geometric parameters (Å, °)*

Cu1···Cu1^{i}	2.994 (2)	N1—C6	1.426 (8)
Cu1···Cu1^{ii}	3.845 (5)	N1—C7	1.319 (8)
Cu1···Cu1^{iii}	3.597 (2)	C1—C2	1.378 (9)
Cu1—O1^{i}	1.950 (4)	C1—C6	1.40 (1)
Cu1···O1^{ii}	2.986 (6)	C2—C3	1.371 (9)
Cu1···O2^{iv}	3.139 (6)	C3—C4	1.37 (1)
Cu1—O1	1.941 (5)	C4—C5	1.40 (1)
Cu1—O2	1.873 (5)	C5—C6	1.400 (9)
Cu1—N1	1.910 (5)	C7—C8	1.412 (9)
Cl1—C4	1.748 (7)	C7—C10	1.495 (9)
O1—C1	1.364 (8)	C8—C9	1.357 (9)
O2—C9	1.300 (8)	C9—C11	1.50 (1)
O1—Cu1—O1^{i}	79.4 (2)	C2—C3—C4	119.6 (7)
O2—Cu1—O1^{i}	100.0 (2)	Cl1—C4—C3	120.3 (6)
N1—Cu1—O1^{i}	161.8 (2)	Cl1—C4—C5	117.7 (6)
Cu1—O1—Cu1^{i}	100.6 (2)	C3—C4—C5	122.0 (7)
O1—Cu1—O2	175.6 (3)	C4—C5—C6	118.2 (7)
O1—Cu1—N1	84.4 (2)	N1—C6—C1	114.3 (6)
O2—Cu1—N1	96.8 (2)	N1—C6—C5	126.1 (7)
Cu1—O1—C1	109.9 (5)	C1—C6—C5	118.9 (7)
Cu1—O2—C9	121.4 (5)	N1—C7—C8	120.6 (6)
Cu1—N1—C6	109.9 (4)	N1—C7—C10	122.6 (6)
Cu1—N1—C7	122.4 (5)	C8—C7—C10	116.8 (6)
C6—N1—C7	126.9 (6)	C7—C8—C9	128.5 (7)
O1—C1—C2	123.0 (7)	O2—C9—C8	125.6 (7)
O1—C1—C6	116.0 (6)	O2—C9—C11	112.3 (6)
C2—C1—C6	121.0 (6)	C8—C9—C11	122.1 (7)
C1—C2—C3	120.1 (7)		

Symmetry codes: (i) $1-x, -y, 2-z$; (ii) $x, 1+y, z$; (iii) $1-x, 1-y, 2-z$; (iv) $x, y-1, z$.

All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically at a distance of 0.95 Å from their parent C atoms, with $B_{iso}(H) = 1.3B_{eq}(C)$. After refining the coordinates of the methyl H atoms for a few cycles, a riding model was used for all H atoms.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976) in *MolEN*. Software used to prepare material for publication: *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1386). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Poly[bis(3,5-dinitrobenzoato-O^1:O^2)-disilver(I)-O^2:*Ag*;*Ag*′:$O^{2\prime}$]

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Abstract

The structure of the title compound, $[Ag_2(C_7H_3N_2O_6)_2]_n$, contains centrosymmetric bis(carboxylato-*O*,*O*′)-bridged silver dimers, which are cross-linked *via* Ag—O(carboxyl) bonds [2.655 (7) Å] to form two stair-like polymeric bands. Within the chelate ring, the Ag—O distances are 2.176 (5) and 2.218 (6) Å, and the Ag···Ag distance is 2.835 (2) Å.

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