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Key indicators

Single-crystal X-ray study

$T = 100$ K

Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å

R factor = 0.027

wR factor = 0.062

Data-to-parameter ratio = 15.5

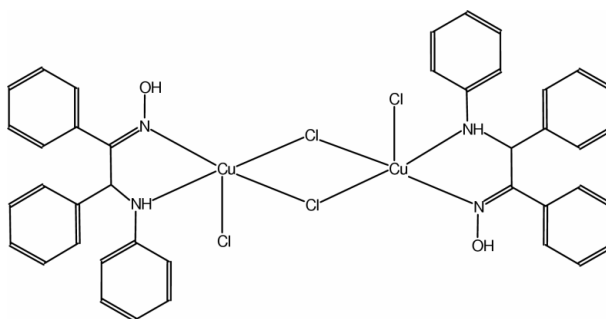
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Di- μ -chloro-bis{chloro[1,2-diphenyl-2-(phenylamino)ethanoximato]copper(II)}

The title compound, $[\text{Cu}_2\text{Cl}_4(\text{C}_{20}\text{H}_{18}\text{N}_2\text{O})_2]$, is a centrosymmetric dimer bridged through the Cl atoms. The bridging Cu_2Cl_2 unit is planar and each Cu^{II} ion has two different distances to the bridging Cl atom. The Cu^{II} ions are coordinated to three Cl and two N atoms in a square-pyramidal disposition. Hydrogen bonds are formed by $-\text{OH}$ and $-\text{NH}$ with the Cl atoms.

Comment

The synthesis and characterization of binuclear copper(II) complexes has made an impact on bio-inorganic chemistry (Solomon *et al.*, 1996). Copper(II) compounds with imidazoles have been used as models to mimic biological systems (Kitajima & Moro-Oka, 1994). Copper(II)-carboxylate adducts with imidazole ligands have a variety of pharmacological properties (Tamura & Imai, 1987). Recently, some attention has been paid to the study of Cu^{II} complexes with $-\text{N}-\text{C}-\text{C}-\text{N}-$, which has a flexible backbone that enables it to act as a bis-chelating ligand (Bernhardt & Shape, 1998; Haidar *et al.*, 1997; Urriaga *et al.*, 1997; Comba *et al.*, 1995). Our study is focused on the determination of the bio-active properties of Cu^{II} complexes with $-\text{N}-\text{C}-\text{C}=\text{N}-$ ligands. While trying to synthesize CuN_4 compounds with that ligand from the reaction of 1,2-diphenyl-2-(phenylamino)ethanoxime with CuCl_2 in ethanol, we obtained a new compound of the type $\text{Cu}_2\text{Cl}_4\text{N}_4$, as shown by the crystallographic study, namely di- μ -chloro-bis{chloro[1,2-diphenyl-2-(phenylamino)ethanoximato]copper(II)}, (I).



(I)

The determination of the crystal structure from data recorded at 100 K shows that (I) consists of binuclear molecules with Cl acting as the bridging atoms (Fig. 1). This compound is centrosymmetric with only one half independent molecule per asymmetric unit. Each metal centre has a square-pyramidal coordination. The four short bonds are to two N atoms, a terminal Cl atom and a bridging Cl atom. The long

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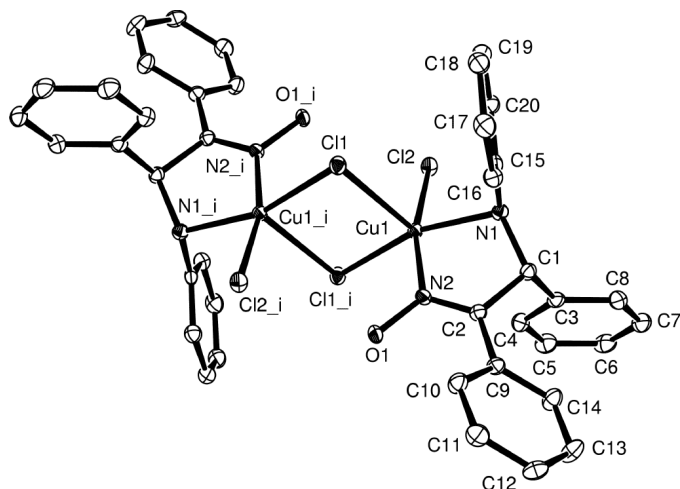


Figure 1

The molecular structure of (I) showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level [symmetry code: (i) $-x, -y, -z$].

apical bond involves the other bridging Cl atom. Thus, in (I) there are two types of Cl atoms, bridging and non-bridging.

The bridging Cu_2Cl_2 unit is planar. The $\text{Cu1}-\text{Cl1}$ and $\text{Cu1}-\text{Cl1}^i$ [symmetry code: (i) $-x, -y, -z$] distances are not equal (Table 1), the larger distance corresponding to the Cl in the apex of the square pyramid and the smaller one to the basal Cl.

The copper-copper distance (Table 1) is similar to the value [3.518 (3) Å] found by Menger *et al.* (1991) in di- μ -chlorobis[chloro(2,9-didodecyl-1,10-phenanthroline-*N,N'*)copper], and it is close to the mean copper-copper distance (3.525 Å) found in the di- μ -chloro pentacoordinated Cu^{II} complexes registered in the April 2001 version of the Cambridge Structural Database (Allen & Kennard, 1993).

The OH group is involved in a bifurcated hydrogen bond (Jeffrey *et al.*, 1985) with Cl1^i and Cl2^i , and $-\text{NH}$ forms an intermolecular hydrogen bond with Cl2^{ii} [symmetry code: (ii) $1-x, -y, -z$] (Table 2).

Experimental

Compound (I) was obtained by slow addition of 30 ml of an absolute ethanol solution (0.001 M) of 1,2-diphenyl-2-phenylamine-ethanoxime to 30 ml of an absolute ethanol solution (0.001 M) of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$. The resulting green precipitate was left overnight, filtered and washed several times with water, followed by ethanol and ether. Recrystallization from absolute ethanol after drying in vacuum gave crystals suitable for X-ray structural analysis; m.p. 456–457 K.

Crystal data

$[\text{Cu}_2\text{Cl}_4(\text{C}_{20}\text{H}_{18}\text{N}_2\text{O})_2]$
 $M_r = 873.61$
 Monoclinic, $P2_1/n$
 $a = 9.1739$ (3) Å
 $b = 22.1696$ (7) Å
 $c = 9.4404$ (3) Å
 $\beta = 104.592$ (1)°
 $V = 1858.07$ (10) Å³
 $Z = 2$

$D_x = 1.561$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 6428 reflections
 $\theta = 2.4$ – 28.5°
 $\mu = 1.47$ mm⁻¹
 $T = 100$ (2) K
 Rhombohedral, dark-green
 $0.42 \times 0.38 \times 0.35$ mm

Data collection

Bruker SMART CCD 1 K area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.842$, $T_{\text{max}} = 1.000$
 21701 measured reflections

4707 independent reflections
 4187 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$
 $\theta_{\text{max}} = 28.5^\circ$
 $h = -12 \rightarrow 12$
 $k = -29 \rightarrow 29$
 $l = -12 \rightarrow 12$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.027$
 $wR(F^2) = 0.063$
 $S = 1.07$
 4707 reflections
 303 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0227P)^2 + 1.7471P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.008$
 $\Delta\rho_{\text{max}} = 0.45 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.40 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

$\text{Cu1}-\text{N2}$	2.0031 (14)	$\text{Cu1}-\text{Cl1}^i$	2.2814 (4)
$\text{Cu1}-\text{N1}$	2.0686 (14)	$\text{Cu1}-\text{Cl1}$	2.6582 (4)
$\text{Cu1}-\text{Cl2}$	2.2371 (4)	$\text{Cu1}-\text{Cu1}^i$	3.5172 (4)
$\text{N2}-\text{Cu1}-\text{N1}$	78.42 (5)	$\text{Cl2}-\text{Cu1}-\text{Cl1}^i$	95.736 (16)
$\text{N2}-\text{Cu1}-\text{Cl2}$	165.92 (4)	$\text{N2}-\text{Cu1}-\text{Cl1}$	93.23 (4)
$\text{N1}-\text{Cu1}-\text{Cl2}$	90.77 (4)	$\text{N1}-\text{Cu1}-\text{Cl1}$	104.52 (4)
$\text{N2}-\text{Cu1}-\text{Cl1}^i$	92.47 (4)	$\text{Cl2}-\text{Cu1}-\text{Cl1}$	98.258 (15)
$\text{N1}-\text{Cu1}-\text{Cl1}^i$	163.53 (4)	$\text{Cl1}^i-\text{Cu1}-\text{Cl1}$	89.528 (15)

Symmetry code: (i) $-x, -y, -z$.

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O1}-\text{H1} \cdots \text{Cl2}^i$	0.84	2.44	3.1516 (12)	143
$\text{O1}-\text{H1} \cdots \text{Cl1}^i$	0.84	2.68	3.2042 (13)	122
$\text{N1}-\text{H2} \cdots \text{Cl2}^{\text{ii}}$	0.83 (2)	2.64 (2)	3.4473 (15)	164.7 (18)

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

All H atoms were located in difference Fourier maps and refined freely except the H atom bonded to O1, which was calculated in an ideal position and refined using a riding model *via* the *SHELXL97* *HFIX* 83 facility.

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SMART-NT*; data reduction: *SAINT-NT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* for Windows (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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