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Di-μ-chloro-bis{chloro[1,2-diphenyl-2-(4-chloro-phenylamino)ethanoximato]copper(II)}

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Key indicators

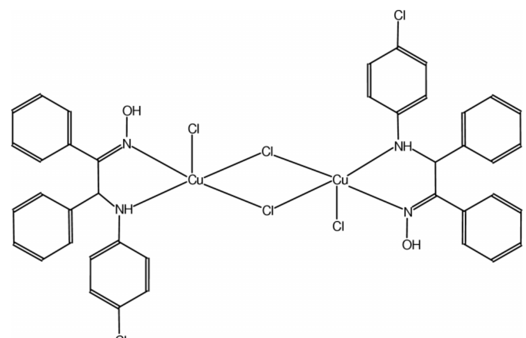
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.023
wR factor = 0.063
Data-to-parameter ratio = 21.1

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

The title compound, $[Cu_2Cl_4(C_{20}H_{17}ClN_2O)_2]$, is a centrosymmetric dimer bridged through the Cl atoms. The bridging Cu_2Cl_2 unit is planar and the geometry around the Cu^{II} ions is that of a square pyramid.

Comment

While studying the bioactive properties of Cu^{II} complexes with different —N—C—C═N— ligands we reported (Puschmann *et al.*, 2001) the results of the crystal structure determination of di-μ-chloro-bis{chloro[1,2-diphenyl-2-(phenylamino)ethanoximato]copper(II)}, (II). In the continuation of this study, we obtained the title compound, (I), by the reaction of 1,2-diphenyl-2-(4-chlorophenylamino)ethanoxime with $CuCl_2$. Compound (I) is similar to (II) but with an additional Cl atom in the phenyl amino group.



(I)

Compound (I) consists of binuclear molecules with Cl acting as the bridging atoms (Fig. 1). There is 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 apical bond involves the other bridging Cl atom.

The bridging Cu_2Cl_2 unit is completely planar. The Cu—Cl1 and Cu—Cl1^{i} [symmetry code: (i) $-x+3/2$, $-y+1/2$, $-z+1$] distances are not equal [2.2454 (3) and 2.6624 (4) Å], the larger distance corresponding to the Cl in the apex of the square pyramid and the smaller one to the basal Cl. The Cl1—Cu—Cl1^{i} angle is larger than that found in (II) [94.306 (11) *versus* 89.528 (15)°], while the Cu—Cl—Cu^{i} angle is smaller than that found in (II) [85.694 (11) *versus* 90.472 (15)°.

The distance between Cu and Cu^{i} is smaller than the value found in (II) [3.3514 (3) *versus* 3.5172 (4) Å] and it is also smaller than 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).

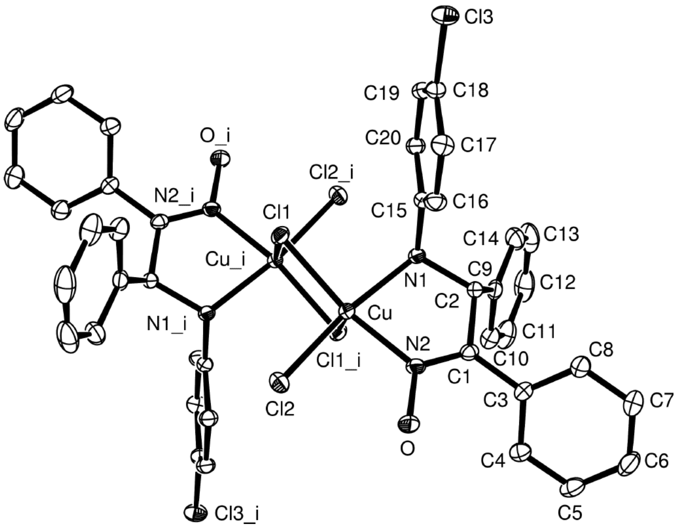


Figure 1
View of (I) showing the atom labelling and 50% probability ellipsoids. [Symmetry code: (i) $-x+3/2$, $-y+1/2$, $-z+1$.]

The position of the OH group with respect to the terminal Cl2 is not the same in (I) and (II). In (I), O and Cl2 point approximately in the same direction and are close enough to form an intramolecular hydrogen bond (Table 2), but in (II), this type of interaction is not possible as the O1 group and Cl2 are pointed in opposite directions. As a measure of this, we compare the torsion angle O—N2—Cu—Cl2 [−4.81 (9)°] with the one found for the equivalent angle (O1—N2—Cu1—Cl2) in (II) [−153.52 (19)°].

Experimental

Compound (I) was obtained by slow addition of 30 ml of an absolute ethanol solution (0.001 *M*) of 1,2-diphenyl-2-(4-chlorophenylamino)ethanoxime to 30 ml of an absolute ethanol solution (0.001 M) of $CuCl_2\cdot2H_2O$. 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. Melting point: 454–455 K.

Crystal data

$[Cu_2Cl_4(C_{20}H_{17}ClN_2O)_2]$
$M_r = 942.49$
Monoclinic, $C2/c$
$a = 20.6420$ (6) Å
$b = 11.5573$ (4) Å
$c = 17.7307$ (5) Å
$\beta = 104.666$ (1)°
$V = 4092.1$ (2) $Å^3$
$Z = 4$
$D_x = 1.530$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8188 reflections
$\theta = 2.2$–28.5°
$\mu = 1.47$ mm^{-1}
$T = 100$ (2) K
Irregular, dark-green
0.46 × 0.42 × 0.34 mm

Data collection

Bruker SMART CCD 1K area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.556$, $T_{max} = 0.606$
23518 measured reflections
5160 independent reflections
4748 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 28.5°$
$h = -27 \rightarrow 27$
$k = -15 \rightarrow 15$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.063$
$S = 1.06$
5160 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0291P)^2 + 3.7353P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu—N2	1.9835 (11)	Cu—Cl2	2.2763 (4)
Cu—N1	2.0692 (11)	Cu—Cl1^{i}	2.6624 (4)
Cu—Cl1	2.2454 (3)	Cu—Cu^{i}	3.3514 (3)
N2—Cu—N1	80.16 (4)	N2—Cu—Cl1^{i}	96.90 (3)
N2—Cu—Cl1	166.27 (3)	N1—Cu—Cl1^{i}	86.25 (3)
N1—Cu—Cl1	92.72 (3)	Cl1—Cu—Cl1^{i}	94.306 (11)
N2—Cu—Cl2	91.07 (3)	Cl2—Cu—Cl1^{i}	98.193 (12)
N1—Cu—Cl2	170.62 (3)	Cu—Cl1—Cu^{i}	85.694 (11)
Cl1—Cu—Cl2	95.170 (13)		

Symmetry code: (i) $\frac{3}{2}-x, \frac{1}{2}-y, 1-z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl1^{i}	0.93	2.87	3.2632 (11)	107
O—H0···Cl2	0.84	2.29	2.9756 (11)	139

Symmetry code: (i) $\frac{3}{2}-x, \frac{1}{2}-y, 1-z$.

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SMART-NT*; data reduction: *SAINT-NT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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