metal-organic papers

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Key indicators

Single-crystal X-ray study T = 295 KMean $\sigma(\text{C}-\text{C}) = 0.005 \text{ Å}$ R factor = 0.043 wR factor = 0.113 Data-to-parameter ratio = 18.5

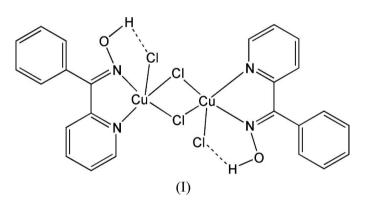
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Di- μ -chloro-bis[chloro(phenyl 2-pyridyl ketone oxime- $\kappa^2 N, N'$)copper(II)]

The title complex, $[Cu_2Cl_4(C_{12}H_{10}N_2O)_2]$, was synthesized by reacting phenyl 2-pyridyl ketone oxime with $CuCl_2 \cdot 2H_2O$ in a basic medium. The organic ligand coordinates to the Cu^{II} ions through its N atoms, while the oxime O atom remains uncoordinated. The Cl^- ions play two roles: one is a terminal ligand and the other is a bridging ligand, the bridge being formed through a crystallographic inversion centre.

Comment

The ligand phenyl 2-pyridyl ketone oxime (*L*1; Taga *et al.*, 1990) is of particular interest for the synthesis of metal– organic complexes, as the hydroxyl group may be deprotonated (Milios *et al.*, 2003). The title compound, (I), is a dinuclear Cu^{II} complex, formulated as $Cu_2(L1)_2Cl_4$. It was synthesized in a basic NaOH medium (see *Experimental*) and its X-ray crystal structure determined.



In compound (I), L1 acts as a bidentate chelating ligand. The asymmetric unit contains a monomeric CuL1Cl₂ moiety with an inversion centre at the mid-point of the Cu···Cu vector. Monomeric units are linked through a long Cu–Cl1 bond (Fig. 1 and Table 1). The resulting coordination geometry for the metallic centre is a distorted square pyramid, with atoms N1, N2, Cl2 and Cl1ⁱ forming the base of the pyramid and atom Cl1 occupying the apical position [symmetry code: (i) -x, 1 - y, -z]. The L1 ligand is bidentate through its pyridine and imine N atoms, while atom O1 does not participate in the coordination. The Cu1–N1 bond is slightly shorter than Cu1–N2 (Table 1). The C1–C6 phenyl ring forms a dihedral angle of 51.9 (1)° with the non-H atoms of the pyridyl/oxime/CuCl₂ group (O1/N1/N2/C7–C12/Cu1/ Cl1ⁱ/Cl2).

The central $(\mu_2$ -Cl)₂Cu₂ four-membered ring is planar by symmetry and the metal···metal separation is 3.442 (1) Å. The OH group forms a short intramolecular hydrogen bond

© 2006 International Union of Crystallography All rights reserved Received 18 July 2006 Accepted 23 August 2006 with atom Cl2 (Table 2), giving a five-membered ring, Cu1/N1/ O1/H1/Cl2 (Fig. 1).

Experimental

The ligand L1 was synthesized according to a reported procedure, as yellow block crystals (Taga et al., 1990). Treatment of a methanolic solution (30 ml) of copper chloride dihydrate (2 mmol, 0.34 g) and L1 (2 mmol, 0.39 g) in the presence of NaOH (4 mmol) led to dark-green block crystals of (I) suitable for X-ray crystal structure analysis. Analysis, found: C 43.26, H 3.10, N 8.50%; calculated for C₂₄H₂₀Cl₄Cu₂N₄O₂: C 43.32, H 3.03, N 8.42%.

Z = 2

 $D_x = 1.676 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

Block, dark green

 $0.32 \times 0.26 \times 0.20 \text{ mm}$

8038 measured reflections

3010 independent reflections 2569 reflections with $I > 2\sigma(I)$

 $w = 1/[\sigma^2(F_0^2) + (0.0569P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

+ 0.7387P]

 $(\Delta/\sigma)_{\rm max} < 0.001$

 $\Delta \rho_{\rm max} = 0.65 \text{ e } \text{\AA}^{-3}$

 $\Delta \rho_{\rm min} = -0.31 \text{ e} \text{ Å}^{-3}$

 $\mu = 2.05 \text{ mm}^{-1}$

T = 295 (2) K

 $R_{\rm int} = 0.022$

 $\theta_{\rm max} = 27.9^\circ$

Crvstal data

 $[Cu_2Cl_4(C_{12}H_{10}N_2O)_2]$ $M_{\rm w} = 665.34$ Monoclinic, $P2_1/c$ a = 103137(14) Å b = 13.8137 (18) Å c = 9.4334 (13) Å $\beta = 101.218 (2)^{\circ}$ V = 1318.3 (3) Å³

Data collection

Bruker APEX area-dectector diffractometer ω and ω scans Absorption correction: multi-scan (SADABS; Bruker, 2002) $T_{\rm min} = 0.533, T_{\rm max} = 0.664$

Refinement

Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.043$ wR(F²) = 0.113 S = 1.083010 reflections 163 parameters H-atom parameters constrained

Table 1

Selected geometric parameters (Å, °).

Cu1-N1	2.001 (2)	Cu1-Cl2	2.2620 (9)
Cu1-N2	2.027 (2)	Cu1-Cl1	2.6977 (9)
Cu1-Cl1 ⁱ	2.2513 (8)		
N1-Cu1-N2	78.15 (10)	Cl1 ⁱ -Cu1-Cl2	94.76 (3)
N1-Cu1-Cl1 ⁱ	171.11 (8)	N1-Cu1-Cl1	94.91 (8)
N2-Cu1-Cl1 ⁱ	96.46 (8)	N2-Cu1-Cl1	92.62 (7)
N1-Cu1-Cl2	89.25 (7)	Cl1 ⁱ -Cu1-Cl1	92.37 (3)
N2-Cu1-Cl2	164.09 (7)	Cl2-Cu1-Cl1	98.16 (3)

Symmetry code: (i) -x, -y + 1, -z.

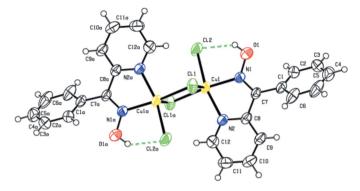


Figure 1

A plot of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as spheres of arbitrary radii. Dashed lines indicate hydrogen bonds. Atoms labelled with the suffix a are at the symmetry position (-x, 1 - y, -z).

Table 2

Hydrogen-bond geometry (Å, °).

$D - H \cdots A$	D-H	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - \mathbf{H} \cdot \cdot \cdot A$
O1−H1···Cl2	0.82	2.32	2.954 (3)	135

H atoms were placed in calculated positions and included in the refinement using a riding-model approximation, with C-H = 0.93 Å and O-H = 0.82 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$.

Data collection: SMART (Bruker, 2002); cell refinement: SMART; data reduction: SAINT (Bruker, 2002); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPII (Johnson, 1976); software used to prepare material for publication: SHELXL97.

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