Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Bis{2-[3-(cyclohexylamino)propyliminomethyl]-4-nitrophenolato}copper(II) diperchlorate

Yu-Xi Sun,[a]* Mei Qin,[a] Ke-Qiang Gu,[a] Guang Yang[b] and Fei Xie[a]

[a]Department of Chemistry, Qufu Normal University, Qufu 273165, People's Republic of China, and [b]Department of Capital Management, Qufu Normal University, Qufu 273165, People's Republic of China

Correspondence e-mail: yuxisun@163.com

Received 17 January 2006
Accepted 23 January 2006
Online 3 February 2006

Key indicators

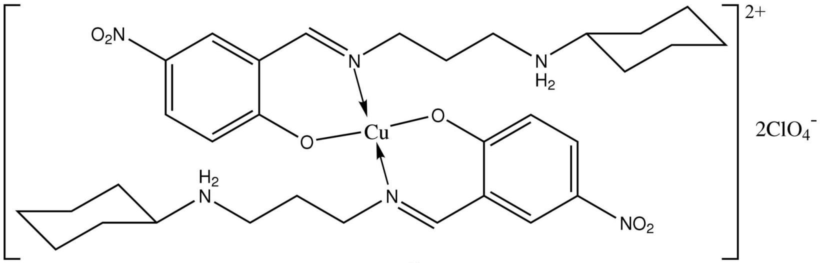
Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.014 Å
Disorder in solvent or counterion
R factor = 0.092
wR factor = 0.115
Data-to-parameter ratio = 16.0

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

The title compound, $[Cu(C_{16}H_{23}N_3O_3)_2](ClO_4)_2$, is a mononuclear copper(II) complex. The Cu^{II} atom is four-coordinated by two imine N atoms and two phenolate O atoms from two Schiff base ligands, forming a slightly distorted square-planar coordination. A perchlorate anion is disordered over two orientations. In the crystal structure, the ions are linked through intermolecular N—H···O hydrogen bonds, forming a three-dimensional network.

Comment

Transition metal compounds are present in the active sites of several important classes of metalloproteins. The study of Schiff base compounds is of great interest in various aspects of chemistry (Downing & Urbach, 1969; Ganeshpure *et al.*, 1996; Bosnich, 1968; Costes *et al.*, 1995). As an extension of work on the structural characterization of Schiff base and copper(II) complexes (Sun, 2005; Sun *et al.*, 2005), a mononuclear copper(II) complex, (I), as a perchlorate salt is reported here.



(I)

The molecular structure of (I) is illustrated in Fig. 1, and selected bond distances and angles are given in Table 1. In the cation of (I), the Cu^{II} atom is four-coordinated by two phenolate O and two imine N atoms from two 2-[3-(cyclohexylamino)propyliminomethyl)-4-nitrophenol ligands. The four coordinating atoms around Cu are approximately coplanar, giving a square-planar coordination with an average deviation of 0.032 (5) Å; the Cu atom lies 0.021 (2) Å above this plane. The two *trans* angles at Cu1 are 179.2 (3) and 177.0 (3)°. The other angles are close to 90°, ranging from 89.2 (2) to 90.8 (2)°, which indicates a slightly distorted square-planar coordination of the Cu atom. The Cu1—O1 and Cu1—O4 bonds(Table 1) are comparable to the corresponding value [1.902 (2) Å] observed in [4-bromo-2-(pyri din-2-yl-methylaminomethyl)phenolato](methanol)copper(II) perchlorate (Sun, 2005) and the value [1.896 (2) Å] in *catena*-poly[[[4-bromo-2-(2-pyridylmethyliminomethyl)phenolato]-copper(II)]-μ-chloro] complex (Sun *et al.*, 2005). However, Cu1—N1 and Cu1—N4 are longer than the corresponding

doi:10.1107/S1600536806002790

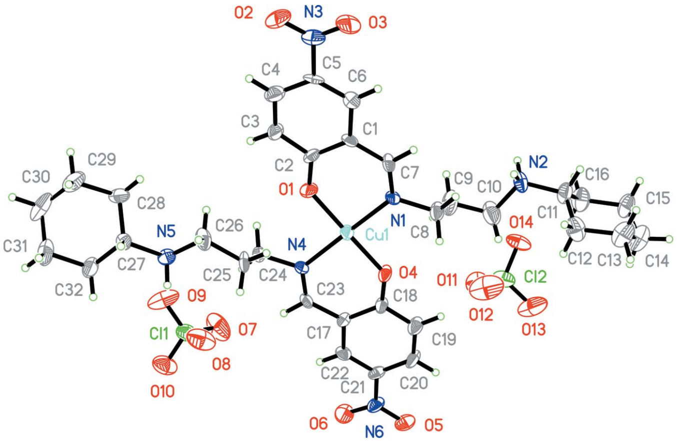


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The minor disorder component is not shown.

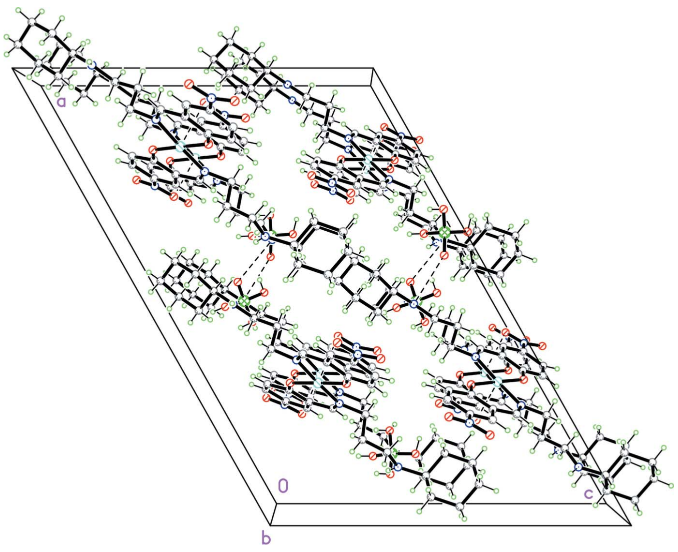


Figure 2
The crystal packing of (I) viewed along the *b* axis. Hydrogen bonds are shown as dashed lines (details are given in Table 2).

values [1.939 (3) Å (Sun, 2005) and 1.961 (3) Å (Sun *et al.*, 2005)].

In the crystal packing, the ions are linked *via* N—H⋯O hydrogen bonds, involving the uncoordinated perchlorate anions and NH_2 groups, to form a three-dimensional network (Table 2 and Fig. 2).

Experimental

For the preparation of the complex, 5-nitrosalicylaldehyde (0.2 mmol, 33.4 mg) and *N*-cyclohexyl-1,3-diaminopropane (0.2 mmol, 31.2 mg) were dissolved in MeOH (10 ml). The mixture was stirred at room temperature for 10 min to give a clear yellow solution, to which was added an aqueous solution (5 ml) of $Cu(ClO_4)_2\cdot6H_2O$ (0.1 mmol, 37.1 mg) with stirring. The mixture was stirred for another 10 min at room temperature and then filtered. The filtrate was set aside fo crystallization. On slow evaporation in air for 13 d, blue block-shaped crystals were formed.

Crystal data

$[Cu(C_{16}H_{23}N_3O_3)_2](ClO_4)_2$
$M_r = 873.19$
Monoclinic, *Cc*
$a = 26.915$ (3) Å
$b = 8.9166$ (11) Å
$c = 18.505$ (2) Å
$\beta = 119.443$ (2)°
$V = 3867.5$ (8) Å^3
$Z = 4$

$D_x = 1.500$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2005 reflections
$\theta = 2.4$–27.7°
$\mu = 0.78$ mm^{-1}
$T = 291$ (2) K
Block, blue
0.25 × 0.16 × 0.03 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.860$, $T_{\max} = 0.977$
15254 measured reflections

8145 independent reflections
3489 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.105$
$\theta_{\max} = 27.5°$
$h = -33 \rightarrow 34$
$k = -11 \rightarrow 11$
$l = -22 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.092$
$wR(F^2) = 0.115$
$S = 0.95$
8145 reflections
509 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0221P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.72$ e Å^{-3}
$\Delta\rho_{\min} = -0.39$ e Å^{-3}
Absolute structure: Flack (1983), 4656 Friedel pairs
Flack parameter: 0.00 (3)

Table 1
Selected geometric parameters (Å, °).

Cu1—O4	1.889 (5)	Cu1—N1	1.991 (6)
Cu1—O1	1.891 (6)	Cu1—N4	2.005 (6)
O4—Cu1—O1	179.2 (3)	O4—Cu1—N4	90.8 (2)
O4—Cu1—N1	89.2 (2)	O1—Cu1—N4	89.6 (3)
O1—Cu1—N1	90.4 (2)	N1—Cu1—N4	177.0 (3)

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N2—H2*A*⋯O11$^{\rm i}$	0.90	2.41	3.147 (14)	139
N2—H2*A*⋯O13$^{\rm i}$	0.90	2.59	3.321 (16)	139
N2—H2*B*⋯O10$^{\rm ii}$	0.90	2.08	2.944 (11)	161
N2—H2*B*⋯O8$^{\rm ii}$	0.90	2.46	3.185 (10)	137
N5—H5*A*⋯O14$^{\rm iii}$	0.90	2.34	2.959 (14)	126
N5—H5*A*⋯O8	0.90	2.66	3.311 (10)	131
N5—H5*B*⋯O5$^{\rm iv}$	0.90	2.48	3.336 (10)	158
N5—H5*B*⋯O9	0.90	2.59	3.209 (10)	127

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

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances of 0.93–0.98 Å and N—H distances of 0.90 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C,N). The O atoms of one of the perchlorate anions are disordered over two sites [occupancies 0.559 (18)/0.441 (18)].

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

The authors thank Qufu Normal University for funding this study.

References

Bosnich, B. (1968). *J. Am. Chem. Soc.* **90**, 627–632.

Bruker (2002). *SMART* (Version 5.628) and *SAINT* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.

Costes, J. P., Dominiguez-Vera, J. M. & Laurent, J. P. (1995). *Polyhedron*, **14**, 2179–2187.

Downing, R. S. & Urbach, F. L. (1969). *J. Am. Chem. Soc.* **91**, 5977–5983.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Ganeshpure, P. A., Tembe, G. L. & Satish, S. (1996). *J. Mol. Catal. A Chem.* **113**, L423–L425.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997*a*). *SHELXL97* and *SHELXS7*. University of Göttingen, Germany.

Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Sun, Y.-X. (2005). *Acta Cryst.* E**61**, m338–m340.

Sun, Y.-X., Gao, Y.-Z., Zhang, H.-L., Kong, D.-S. & Yu, Y. (2005). *Acta Cryst.* E**61**, m1055–m1057.