Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Dichloro[*trans*-(1*R*,2*R*)-*N*,*N*,*N*′,*N*′-tetramethylcyclohexane-1,2-diamine]copper(II)

Svitlana V. Pavlova, Yuh-Sheng Wen and Sunney I. Chan*

Institute of Chemistry, Academia Sinica, Nankang, Taipei, Taiwan

Correspondence e-mail: chans@chem.sinica.edu.tw

Key indicators

Single-crystal X-ray study
T = 300 K
Mean σ(C–C) = 0.012 Å
R factor = 0.039
wR factor = 0.151
Data-to-parameter ratio = 18.4

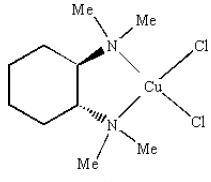
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 11 July 2003
Accepted 4 August 2003
Online 23 August 2003

In the crystal structure of the title compound, $[CuCl_2(C_{10}H_{22}N_2)]$, the Cu^{II} atom adopts a distorted square-planar geometry, the basal plane of which is formed by two N atoms from the cyclohexanediamine ligand, with Cu—N distances of 2.052 (7) Å, and two Cl atoms with Cu—Cl distances of 2.247 (2) Å. A twofold axis of symmetry passes through the Cu atom, such that all other atoms are symmetry-related in pairs. The torsion angle of C—N—Cu—Cl is 174.9 (3)°. The cyclohexane ring is in a chair form.

Comment

Copper(I) complexes with biologically relevant amine or mixed pyridyl/amine donor ligands have attracted much attention in recent years as simple models for the biomimetic chemistry of copper-containing proteins, which bind or activate dioxygen (Tolman, 1997; Schindler, 2000; Solomon *et al.*, 2001). *trans*-(1*R*,2*R*)-*N*,*N*,*N*′,*N*′-Tetramethylcyclohexane-1,2-diamine (*L*) is of interest because of its preorganized nature for binding a single metal and its chirality. Under argon conditions, *L* stabilizes copper(I) as a mononuclear trigonal-planar complex, formulated as $[L\mathrm{Cu}(CH_3CN)](\mathrm{OTf})$ (OTf is trifluoromethanesulfonate), which was isolated but has not been structurally characterized (Cole *et al.*, 1996). Our attempts to isolate the $[L\mathrm{Cu}(CH_3CN)]ClO_4$ complex from the reaction of *L* with $[Cu(CH_3CN)_4]ClO_4$, resulted in a redox reaction which produced the title compound, (I), as a major product. The molecular structure of (I) is shown in Fig. 1.



(I)

The copper(II) metal center resides on a twofold rotation axis and displays a distorted square-planar coordination geometry. The Cu—Cl bond length is 2.247 (2) Å and Cu—N is 2.052 (7) Å. The bond angles at the Cu center are: N—Cu—N^i 83.9 (4)°, N—Cu—Cl 93.4 (2)°, N—Cu—Cl^i 170.6 (3)° and Cl—Cu—Cl^i 90.7 (2)° [symmetry code (i): $2-x$, $-y$, $2-z$]. The torsion angle C1—N—Cu—Cl is 174.9 (3)°. The unit cell contains two symmetry-related molecules of (I). According to the interatomic distances there are no hydrogen bonds between adjacent molecules.

Experimental

trans-(1*R*,2*R*)-*N*,*N*,*N*′,*N*′-Tetramethylcyclohexane-1,2-diamine was synthesized from commercially available (1*R*)-*trans*-1,2-cyclohexanediamine (Aldrich), using the Eschweiler–Clark methylation of amines method (Remenar *et al.*, 1997). $[Cu(CH_3CN)_4]ClO_4$ was prepared according to a published procedure (Gill *et al.*, 1995). Equimolar amounts of $[Cu(CH_3CN)_4]ClO_4$ (0.05 g, 0.15 mmol) and *L* (0.028 ml, 0.15 mmol) were combined in 10 ml of a dry CH_3CN/CH_2Cl_2 (3:1, *v*/*v*) mixture under argon. The resulting colorless solution turned blue–green, while stirring for 1 d at room temperature under argon. The products were precipitated with 50 ml of dry diethyl ether to give 0.30 g (66%) of a blue–green powder. Recrystallization from a dichloromethane/acetonitrile (4:1, *v*/*v*) mixture, followed by slow diffusion of cyclohexane into a CH_3CN solution, afforded dark-blue crystals of the title compound, (I).

Crystal data

$[CuCl_2(C_{10}H_{22}N_2)]$
M_r = 304.74
Monoclinic, *C*2
a = 8.8574 (8) Å
b = 10.595 (1) Å
c = 8.0634 (5) Å
β = 118.939 (6)°
V = 662.2 (1) Å^3
Z = 2
D_x = 1.528 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 24 reflections
θ = 8.7–17.1°
μ = 2.03 mm^{-1}
T = 300 (2) K
Prism, dark blue
0.44 × 0.31 × 0.25 mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.481, T_{max} = 0.603
1379 measured reflections
1289 independent reflections
1225 reflections with $I > 2\sigma(I)$
R_{int} = 0.09
θ_{max} = 25.9°
h = −10 → 9
k = −12 → 12
l = 0 → 9
2 standard reflections
frequency: 60 min
intensity decay: 4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.151
S = 1.02
1289 reflections
70 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0748P)^2 + 6.5193P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}
Absolute structure: Flack (1983), 607 Friedel pairs
Flack parameter = −0.04 (5)

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); 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: *WinGX* (Farrugia, 1999).

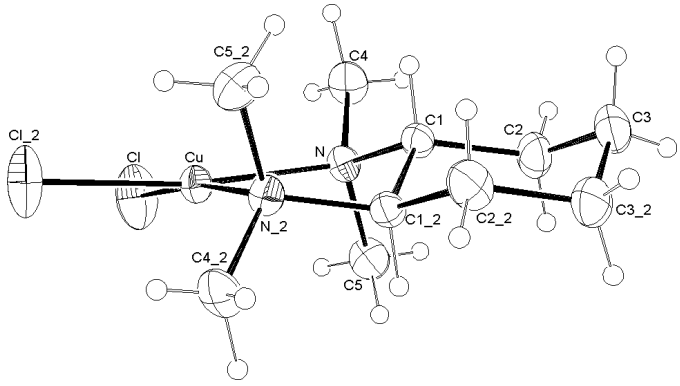


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids, showing the atom-numbering scheme employed. H atoms are shown as small spheres of the arbitrary radii. The suffix _2 corresponds to symmetry code (i) in the *Comment* text.

This work was supported by a grant from the National Science Council (NSC-90-2113-M-001-080) of the Republic of China.

References

Cole, A. P., Root, D. E., Mukherjee, P., Solomon, E. I. & Stack, T. D. P. (1996). *Science*, **273**, 1848–1850.
Enraf–Nonius (1994). *CAD*-4 *EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Gill, D. S., Rodehüser, L., Rubini, P. & Delpuech, J.-J. (1995). *J. Chem. Soc. Faraday Trans.* **91**, 2307–2312.
Harms, K. & Wocadlo, S. (1996). *XCAD*4. University of Marburg, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Remenar, J. F., Lucht, B. L. & Collum, D. B. (1997). *J. Am. Chem. Soc.* **119**, 5567–5572.
Schindler, S. (2000). *Eur. J. Inorg. Chem.* pp. 2311–2326.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Solomon, E. I., Chen, P., Metz, M., Lee, S.-K. & Palmer, A. E. (2001). *Angew. Chem. Int. Ed. Engl.* **40**, 4570–4590.
Tolman, W. B. (1997). *Acc. Chem. Res.* **30**, 227–237.