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Dichloro[(1*R*,2*R*)-*N*-(2-pyridylmethylene)-1,2-cyclohexanediamine]copper(II)

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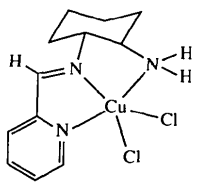
Abstract

The crystal structure of $[CuCl_2(C_{12}H_{17}N_3)]$, containing a five-coordinate Cu^{II} atom with distorted trigonal-bipyramidal coordination, is reported. The absolute configuration (1*R*,2*R*) has been verified.

Comment

We are interested in the synthesis and the structural chemistry of Cu complexes containing multidentate ligands (Wong, Gao & Wong, 1993). The tridentate ligand (1*R*,2*R*)-*N*-(2-pyridylmethylene)-1,2-cyclohexanediamine, containing three different types of N donor atoms, was prepared by condensation of an equimolar mixture of 2-pyridinecarboxaldehyde and (1*R*,2*R*)-1,2-diaminocyclohexane (L_1).



(I)

An *ORTEP*II plot (Johnson, 1976) of the molecule, (I), is shown in Fig. 1. The Cu atom has a distorted trigonal-bipyramidal environment, consisting of two Cl and three N atoms of the ligand L_1. An increase in the Cu—N distances from Cu—N(imine) 1.983 (4), through Cu—$N(NH_2)$ 2.020 (4) to Cu—N(pyridine) 2.064 (4) Å, was observed. The Cu—Cl distances are significantly different [2.295 (1) and 2.396 (1) Å]. The cyclohexyl ring defined by C(1)–C(6) is in a chair conformation.

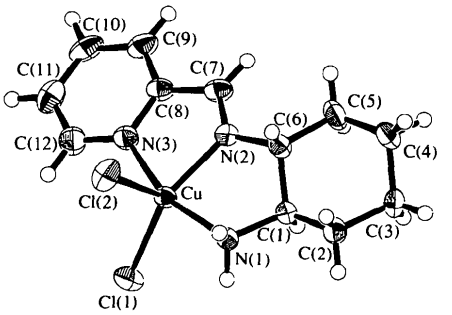


Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of the molecule with 50% probability ellipsoids showing the numbering scheme. H atoms are shown as spheres of arbitrary radii.

Experimental

The title compound was prepared by treating $CuCl_2$ with an equivalent amount of L_1 in methanol. The reaction mixture was heated under reflux for 30 min. The solvent was removed under vacuum to give a blue residue which was redissolved in a minimum amount of water. Slow evaporation of the aqueous solution (2–3 d) at room temperature afforded blue crystals suitable for X-ray analysis.

Crystal data

$[CuCl_2(C_{12}H_{17}N_3)]$	Mo $K\alpha$ radiation
M_r = 337.83	λ = 0.71073 Å
Orthorhombic	Cell parameters from 25 reflections
$P2_12_12_1$	θ = 10–14°
a = 8.235 (2) Å	μ = 1.95 mm^{-1}
b = 8.602 (1) Å	T = 293 K
c = 19.604 (1) Å	Block
V = 1388.7 (3) Å^3	0.34 × 0.30 × 0.28 mm
Z = 4	Blue
D_x = 1.615 Mg m^{-3}	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.016
ω–2θ scans	θ_{max} = 25°
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)	$h = 0 \rightarrow 9$
T_{min} = 0.845, T_{max} = 0.998	$k = 0 \rightarrow 10$
2906 measured reflections	$l = 0 \rightarrow 22$
2459 independent reflections	3 standard reflections
2201 observed reflections [$F_o > 3\sigma(F_o)$]	frequency: 120 min
	intensity decay: <2%

Refinement

Refinement on F	$w = 4F_o^2/[\sigma^2(F_o^2) + 0.04(F_o^2)^2]$
R = 0.031	$(\Delta/\sigma)_{max}$ = 0.02
wR = 0.042	

S = 1.034
2201 reflections
163 parameters
H-atom parameters not refined

$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.59 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Cu	0.76554 (6)	0.60628 (7)	0.28950 (3)	2.270 (9)
Cl(1)	0.6371 (2)	0.7995 (2)	0.34955 (6)	3.19 (2)
Cl(2)	0.6713 (2)	0.3591 (2)	0.32963 (7)	3.52 (2)
N(1)	0.6179 (4)	0.6078 (5)	0.2072 (2)	2.51 (7)
N(2)	0.9339 (5)	0.5658 (5)	0.2194 (2)	2.50 (8)
N(3)	0.9736 (5)	0.6123 (5)	0.3481 (2)	2.63 (7)
C(1)	0.7199 (6)	0.6415 (5)	0.1448 (2)	2.53 (9)
C(2)	0.6326 (6)	0.6140 (7)	0.0786 (2)	3.2 (1)
C(3)	0.7492 (9)	0.6596 (6)	0.0187 (2)	3.6 (1)
C(4)	0.9056 (7)	0.5631 (7)	0.0226 (3)	3.4 (1)
C(5)	0.9940 (7)	0.5808 (8)	0.0933 (3)	3.6 (1)
C(6)	0.8740 (6)	0.5430 (6)	0.1495 (2)	2.48 (9)
C(7)	1.0822 (6)	0.5650 (6)	0.2370 (3)	2.9 (1)
C(8)	1.1109 (5)	0.5885 (6)	0.3116 (2)	2.48 (9)
C(9)	1.2625 (6)	0.5901 (6)	0.3408 (3)	3.4 (1)
C(10)	1.2732 (6)	0.6081 (7)	0.4114 (3)	3.8 (1)
C(11)	1.1310 (8)	0.6256 (7)	0.4485 (3)	3.8 (1)
C(12)	0.9851 (6)	0.6300 (7)	0.4156 (3)	3.2 (1)

Table 2. *Selected geometric parameters* (Å, °)

Cu—Cl(1)	2.295 (1)	C(1)—C(6)	1.528 (7)
Cu—Cl(2)	2.396 (1)	C(2)—C(3)	1.568 (7)
Cu—N(1)	2.020 (4)	C(3)—C(4)	1.534 (9)
Cu—N(2)	1.983 (4)	C(4)—C(5)	1.572 (7)
Cu—N(3)	2.064 (4)	C(5)—C(6)	1.516 (8)
N(1)—C(1)	1.512 (6)	C(7)—C(8)	1.496 (8)
N(2)—C(6)	1.469 (6)	C(8)—C(9)	1.374 (7)
N(2)—C(7)	1.269 (6)	C(9)—C(10)	1.394 (8)
N(3)—C(8)	1.353 (6)	C(10)—C(11)	1.386 (9)
N(3)—C(12)	1.335 (7)	C(11)—C(12)	1.365 (9)
C(1)—C(2)	1.503 (7)		
Cl(1)—Cu—Cl(2)	108.97 (5)	N(1)—C(1)—C(6)	107.8 (4)
Cl(1)—Cu—N(1)	97.3 (1)	C(2)—C(1)—C(6)	111.2 (4)
Cl(1)—Cu—N(2)	143.7 (1)	C(1)—C(2)—C(3)	108.4 (4)
Cl(1)—Cu—N(3)	94.5 (1)	C(2)—C(3)—C(4)	110.0 (4)
Cl(2)—Cu—N(1)	94.2 (1)	C(3)—C(4)—C(5)	112.4 (4)
Cl(2)—Cu—N(2)	107.3 (1)	C(4)—C(5)—C(6)	108.6 (4)
Cl(2)—Cu—N(3)	96.2 (1)	N(2)—C(6)—C(1)	105.2 (4)
N(1)—Cu—N(2)	82.4 (2)	N(2)—C(6)—C(5)	115.5 (5)
N(1)—Cu—N(3)	160.7 (1)	C(1)—C(6)—C(5)	112.2 (5)
N(2)—Cu—N(3)	79.0 (2)	N(2)—C(7)—C(8)	114.6 (4)
Cu—N(1)—C(1)	108.2 (3)	N(3)—C(8)—C(7)	113.9 (4)
Cu—N(2)—C(6)	115.8 (3)	N(3)—C(8)—C(9)	122.5 (4)
Cu—N(2)—C(7)	119.0 (3)	C(7)—C(8)—C(9)	123.5 (4)
C(6)—N(2)—C(7)	125.2 (4)	C(8)—C(9)—C(10)	118.2 (5)
Cu—N(3)—C(8)	113.3 (3)	C(9)—C(10)—C(11)	118.6 (5)
Cu—N(3)—C(12)	127.8 (3)	C(10)—C(11)—C(12)	119.9 (5)
C(8)—N(3)—C(12)	118.8 (5)	N(3)—C(12)—C(11)	121.9 (5)
N(1)—C(1)—C(2)	113.8 (4)		

The structure was solved by Patterson methods and refined by full-matrix least squares. H atoms were generated in idealized positions. The absolute configuration, (1*R*,2*R*) as in the chiral diamine used in the preparation, has been checked by refinement (R = 0.031 instead of 0.044 for the enantiomeric structure) based on the anomalous dispersion of the Cu and Cl atoms. All calculations were performed using the *Structure Determination Package* (Enraf–Nonius, 1985) on a MicroVAX II computer.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1170). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(cupferronato)copper(II), $[Cu(C_6H_5N_2O_2)_2]$

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Abstract

The Cu atom in the title molecule, bis(*N*-nitroso-*N*-phenylhydroxylaminato-*O*,*O*′)copper(II), is coordinated by four donor O atoms to form a planar, almost square coordination geometry. The O1—Cu—O2 and O1—Cu—O2*A* angles are 81.77 (9) and 98.23 (9)°, respectively, and the Cu1—O1 and Cu1—O2 bond lengths are 1.902 (2) and 1.892 (2) Å, respectively.