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catena-Poly[[chlorido[(1*S*,2*S*)-cyclohexane-1,2-diamine-$\kappa^2 N,N'$]copper(II)]-μ_3-chlorido]

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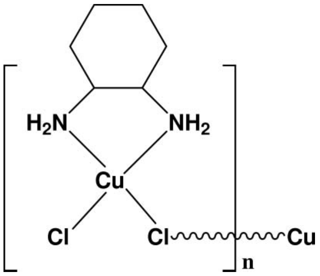
Key indicators: single-crystal X-ray study; *T* = 296 K; mean σ(C–C) = 0.005 Å; *R* factor = 0.031; *wR* factor = 0.082; data-to-parameter ratio = 18.8.

In the repeat unit of the title compound, $[CuCl_2(C_6H_{14}N_2)]$, there are two chiral C atoms with an *S* configuration. In the crystal packing, one Cl atom plays an important role, acting as a bridge linking neighboring units, forming a polymeric one-dimensional ladder-like structure. The Cu atom has an approximately square-planar primary coordination geometry, with the bridging Cl atoms forming much longer bonds in the axial positions.

Related literature

For related literature, see: Choi *et al.* (1999); Khokhar *et al.* (1993); Spingler *et al.* (2001); Pavlova *et al.* (2003).



Experimental

Crystal data

$[CuCl_2(C_6H_{14}N_2)]$
M_r = 248.63
Orthorhombic, $P2_12_12_1$
a = 5.704 (1) Å
b = 6.819 (1) Å
c = 24.823 (4) Å
V = 965.5 (3) Å^3
Z = 4
Mo *Kα* radiation
μ = 2.76 mm^{-1}
T = 296 (2) K
0.20 × 0.20 × 0.12 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.58, T_{max} = 0.72
5714 measured reflections
1884 independent reflections
1766 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.082
S = 1.02
1884 reflections
100 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.69 e Å^{-3}
$\Delta\rho_{min}$ = −0.55 e Å^{-3}
Absolute structure: Flack (1983), 744 Friedel pairs
Flack parameter: 0.011 (19)

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2011).

References

Bruker (2000). *SMART* (Version 5.625), *SAINT* (Version 6.01), *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
Choi, K. Y., Kim, Y. J., Ryu, H. & Suh, I. H. (1999). *Inorg. Chem. Commun.* **2**, 176–180.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Khokhar, A. R., Xu, Q. & Al-Baker, S. (1993). *J. Inorg. Biochem.* **52**, 51–58.
Pavlova, S. V., Wen, Y.-S. & Chan, S. I. (2003). *Acta Cryst.* E**59**, m792–m793.
Spingler, B., Whittington, D. A. & Lippard, S. J. (2001). *Inorg. Chem.* **40**, 5596–5602.

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supplementary materials

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catena-Poly[[chlorido[(1*S*,2*S*)-cyclohexane-1,2-diamine-κ^2*N*,*N*']copper(II)]-μ_3-chlorido]

D.-W. Fu and H. Zhao

Comment

cis-Diamminedichloroplatinum(II) received FDA approval in 1979 for use as an anticancer drug (Spingler, *et al.*, 2001). Since then, scientists have paid much attention to coordination compounds including diamino coordination sites. Diaminocyclohexane is one of the ligands strongly attracting scientists (Khokhar, *et al.*, 1993). A number of coordination compounds were synthesized by using diaminocyclohexane reacting with all kinds of metals (Choi, *et al.*, 1999). Furthermore, diaminocyclohexane is an excellent chiral source, so its coordination compounds are broadly applied in catalysis. Herein we describe the crystal structure of title compound(I).

The molecule of the title complex, (I) (Fig. 1), is unsymmetrical. The Cu atoms in (I) are coordinated by the two amino groups of the organic ligand as well as by two chloro ligands in an almost square planar geometry. The bond lengths and angles in (I) are within normal ranges (Pavlova, *et al.*, 2003). In (I) the Cu1–N1 and Cu1–N2 bond lengths of 2.006 (2) and 2.016 (2) Å are slightly shorter than that of Dichloro-(*trans*-(1*R*,2*R*)-*N*,*N*,*N*',*N*'-tetramethylcyclohexane-1,2-diamine)-copper(ii) (II) (Pavlova, *et al.*, 2002), but the Cu1–Cl1 and Cu1–Cl2 bond lengths of 2.3047 (8) and 2.2832 (8) Å are slightly longer than that of (II). This is considered to be caused by the different packing type and different coordination mode of the chloro ligands. In the molecule there are two chiral carbon atoms C1 and C6 both adopting S configuration. In the crystal packing the Cl1 atom plays an important role acting as a bridge linking neighboring molecular units to form a polymeric one-dimensional ladderlike structure (Fig.2). The corresponding copper chlorine bond lengths are significantly longer than the bonds described above leading to a highly Jahn–Teller distorted octahedral coordination mode of the copper atoms in the crystal structure of (I).

Experimental

Under solvothermal conditions, the reaction of (1*S*,2S)-1,2-diaminocyclohexane (0.0228 g, 0.2 mmol) with copper chloride (0.0347 g, 0.2 mmol) in a mixture of methanol and hydrochloric acid (2 ml, volume ratio: 10:1) at 1173 K for one day afforded square blue crystals of title compound.

Refinement

All carbon-bound H atoms were positioned geometrically, with C—H = 0.97 Å and included in the refinement as riding, with U_{iso}(H) = 1.2U_{eq}(C). The H atoms attached to N were visible in the difference Fourier map and were subsequently treated as riding atoms, with N—H = 0.90 Å, and with U_{iso}(H) = 1.2U_{eq} (N).

Figures

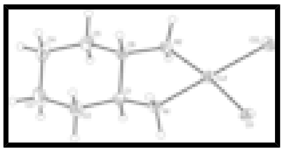

Fig. 1. The structure of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

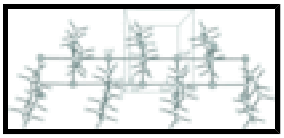

Fig. 2. View, approximately down the *c* axis, of the one-dimensional-dimension ladderlike structure. Symmetry code: (i) −1/2 + *x*,1/2 − *y*,-*z* (ii) 1/2 + *x*,1/2 − *y*,-*z*

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Crystal data

$[CuCl_2(C_6H_{14}N_2)]$	$F_{000} = 508$
$M_r = 248.63$	$D_x = 1.711$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 875 reflections
$a = 5.704$ (1) Å	θ = 2.1–25.2º
$b = 6.819$ (1) Å	$\mu = 2.76$ mm^{-1}
$c = 24.823$ (4) Å	$T = 296$ (2) K
$V = 965.5$ (3) Å^3	Square, blue
$Z = 4$	0.20 × 0.20 × 0.12 mm

Data collection

Bruker SMART APEX CCD diffractometer	1884 independent reflections
Radiation source: sealed tube	1766 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.044$
$T = 296(2)$ K	$\theta_{\text{max}} = 26.0$º
φ and ω scans	$\theta_{\text{min}} = 3.1$º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -6 \rightarrow 7$
$T_{\text{min}} = 0.58$, $T_{\text{max}} = 0.72$	$k = -8 \rightarrow 8$
5714 measured reflections	$l = -30 \rightarrow 30$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.031$	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.082$	$(\Delta/\sigma)_{\text{max}} < 0.001$

$S = 1.03$	$\Delta\rho_{max} = 0.69$ e Å^{-3}
1884 reflections	$\Delta\rho_{min} = -0.55$ e Å^{-3}
100 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 744 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.011 (19)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.3284 (6)	0.4129 (4)	−0.12770 (12)	0.0275 (7)
H1	0.4965	0.4423	−0.1264	0.033*
C2	0.2723 (9)	0.3424 (5)	−0.18417 (11)	0.0435 (9)
H2A	0.1097	0.3011	−0.1858	0.052*
H2B	0.3698	0.2301	−0.1928	0.052*
C3	0.3145 (9)	0.5042 (6)	−0.22536 (13)	0.0506 (12)
H3A	0.4810	0.5325	−0.2272	0.061*
H3B	0.2647	0.4593	−0.2606	0.061*
C4	0.1835 (8)	0.6890 (6)	−0.21115 (13)	0.0455 (10)
H4A	0.0163	0.6647	−0.2135	0.055*
H4B	0.2227	0.7909	−0.2369	0.055*
C5	0.2436 (8)	0.7590 (4)	−0.15455 (11)	0.0356 (7)
H5A	0.4074	0.7969	−0.1531	0.043*
H5B	0.1496	0.8732	−0.1458	0.043*
C6	0.1980 (5)	0.5989 (4)	−0.11358 (12)	0.0265 (7)
H6	0.0300	0.5690	−0.1146	0.032*
Cl1	0.26572 (14)	0.09330 (9)	0.02474 (3)	0.02790 (17)
Cl2	0.26008 (16)	0.55471 (10)	0.06844 (3)	0.03167 (19)
Cu1	0.26675 (7)	0.40155 (4)	−0.013331 (12)	0.02443 (14)
N1	0.2789 (6)	0.2686 (3)	−0.08535 (8)	0.0272 (6)
H1A	0.3915	0.1761	−0.0852	0.033*
H1B	0.1408	0.2095	−0.0920	0.033*
N2	0.2584 (6)	0.6497 (3)	−0.05747 (9)	0.0258 (5)
H2C	0.1508	0.7327	−0.0439	0.031*
H2D	0.3992	0.7091	−0.0564	0.031*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0276 (17)	0.0274 (15)	0.0276 (15)	0.0006 (14)	0.0007 (12)	0.0000 (12)
C2	0.063 (3)	0.0399 (17)	0.0281 (16)	−0.001 (2)	0.000 (2)	−0.0057 (13)
C3	0.070 (3)	0.054 (2)	0.0271 (16)	−0.003 (2)	0.0053 (18)	−0.0010 (15)
C4	0.051 (3)	0.053 (2)	0.0328 (17)	−0.0051 (19)	−0.0067 (17)	0.0146 (16)
C5	0.041 (2)	0.0326 (15)	0.0333 (14)	0.003 (2)	0.001 (2)	0.0071 (12)
C6	0.0227 (16)	0.0284 (15)	0.0283 (14)	0.0002 (14)	−0.0010 (12)	0.0013 (11)
Cl1	0.0211 (4)	0.0254 (3)	0.0372 (4)	0.0006 (4)	0.0011 (3)	0.0069 (2)
Cl2	0.0316 (4)	0.0339 (4)	0.0296 (3)	−0.0027 (4)	0.0000 (4)	−0.0052 (3)
Cu1	0.0261 (2)	0.0227 (2)	0.0245 (2)	0.00011 (19)	−0.00035 (16)	0.00093 (11)
N1	0.0291 (16)	0.0222 (11)	0.0303 (12)	0.0012 (13)	−0.0017 (13)	−0.0018 (9)
N2	0.0269 (14)	0.0247 (11)	0.0259 (11)	0.0005 (13)	0.0020 (13)	−0.0030 (9)

Geometric parameters (Å, °)

C1—N1	1.467 (4)	C5—C6	1.514 (4)
C1—C6	1.512 (4)	C5—H5A	0.9700
C1—C2	1.516 (4)	C5—H5B	0.9700
C1—H1	0.9800	C6—N2	1.476 (4)
C2—C3	1.524 (5)	C6—H6	0.9800
C2—H2A	0.9700	Cl1—Cu1	2.3047 (8)
C2—H2B	0.9700	Cl2—Cu1	2.2832 (8)
C3—C4	1.507 (6)	Cu1—N1	2.006 (2)
C3—H3A	0.9700	Cu1—N2	2.016 (2)
C3—H3B	0.9700	N1—H1A	0.9000
C4—C5	1.523 (4)	N1—H1B	0.9000
C4—H4A	0.9700	N2—H2C	0.9000
C4—H4B	0.9700	N2—H2D	0.9000

N1—C1—C6	107.6 (2)	C6—C5—H5B	109.5
N1—C1—C2	114.1 (3)	C4—C5—H5B	109.5
C6—C1—C2	112.1 (3)	H5A—C5—H5B	108.1
N1—C1—H1	107.6	N2—C6—C1	107.5 (2)
C6—C1—H1	107.6	N2—C6—C5	115.1 (2)
C2—C1—H1	107.6	C1—C6—C5	111.4 (3)
C1—C2—C3	110.9 (3)	N2—C6—H6	107.5
C1—C2—H2A	109.5	C1—C6—H6	107.5
C3—C2—H2A	109.4	C5—C6—H6	107.5
C1—C2—H2B	109.4	N1—Cu1—N2	84.03 (9)
C3—C2—H2B	109.4	N1—Cu1—Cl2	178.92 (9)
H2A—C2—H2B	108.0	N2—Cu1—Cl2	95.67 (6)
C4—C3—C2	111.7 (3)	N1—Cu1—Cl1	87.31 (7)
C4—C3—H3A	109.3	N2—Cu1—Cl1	171.15 (7)
C2—C3—H3A	109.3	Cl2—Cu1—Cl1	93.02 (3)
C4—C3—H3B	109.3	C1—N1—Cu1	109.99 (18)
C2—C3—H3B	109.3	C1—N1—H1A	109.7

H3A—C3—H3B	107.9	Cu1—N1—H1A	109.7
C3—C4—C5	111.5 (3)	C1—N1—H1B	109.7
C3—C4—H4A	109.3	Cu1—N1—H1B	109.7
C5—C4—H4A	109.3	H1A—N1—H1B	108.2
C3—C4—H4B	109.3	C6—N2—Cu1	108.75 (16)
C5—C4—H4B	109.3	C6—N2—H2C	109.9
H4A—C4—H4B	108.0	Cu1—N2—H2C	109.9
C6—C5—C4	110.8 (3)	C6—N2—H2D	109.9
C6—C5—H5A	109.5	Cu1—N2—H2D	109.9
C4—C5—H5A	109.5	H2C—N2—H2D	108.3

Fig. 1

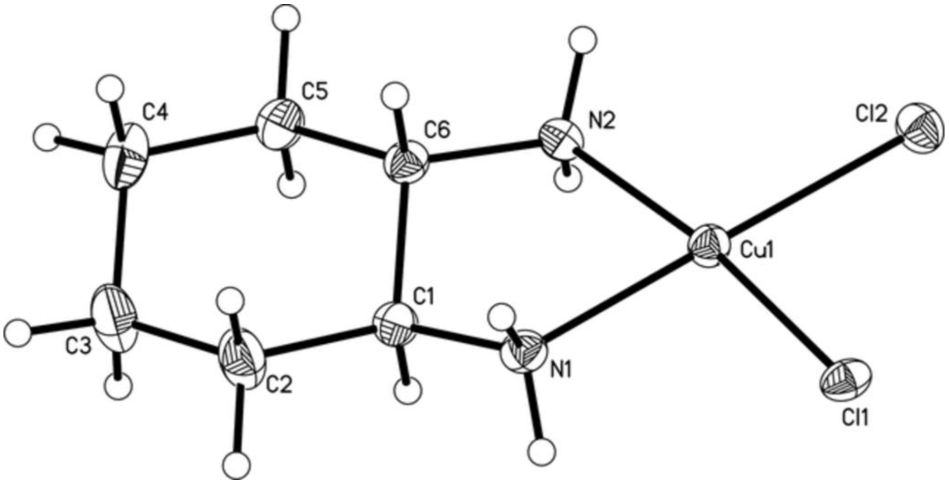

Fig. 2