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[2,6-Bis(*p*-tolyliminomethyl)pyridine-κ^3N,N',N'']dichloridocopper(II)

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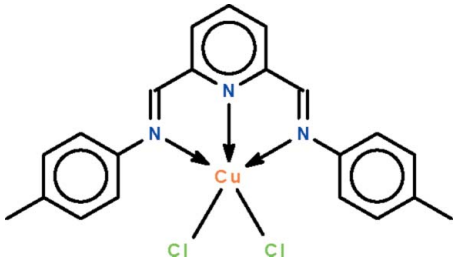
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.030; wR factor = 0.070; data-to-parameter ratio = 17.5.

The title compound, $[CuCl_2(C_{21}H_{19}N_3)]$, lies on a twofold rotation axis that passes through the $N_{pyridyl}$—Cu bond; this symmetry element relates one half of the organic ligand to the other as well as one Cl ligand to the other. The three N atoms span the axial–equatorial–axial sites of the trigonal-bipyramidal coordination polyhedron; the geometry of the Cu^{II} atom is 31% distorted from trigonal-bipyramidal (towards square-pyramidal along the Berry pseudorotation pathway).

Related literature

For a chromium chloride adduct with a similar ligand, see: Li *et al.* (2010).



Experimental

Crystal data

$[CuCl_2(C_{21}H_{19}N_3)]$
M_r = 447.83
Orthorhombic, *Fdd*2
a = 11.5220 (13) Å
b = 35.522 (4) Å
c = 9.327 (1) Å
V = 3817.4 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.44 mm^{-1}
T = 100 K
0.36 × 0.12 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.626, T_{max} = 0.972
8753 measured reflections
2190 independent reflections
2023 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.070
S = 1.04
2190 reflections
125 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Absolute structure: Flack (1983), 858 Friedel pairs
Flack parameter: 0.014 (14)

Table 1
Selected bond lengths (Å).

Cu1—N1	1.968 (3)	Cu1—Cl1	2.3187 (7)
Cu1—N2	2.101 (2)		

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5030).

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supporting information

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S1. Comment

A recent study reported the chromium(III) chloride adduct of 2,6-bis(*p*-bromphenylimino)pyridine; the *N*-heterocycle chelates to the metal atom in a terdentate manner (Li *et al.*, 2010). The copper dichlroide adduct of 2,6-bis(*p*-tolylimino)-pyridine adopts a similar structure. The $CuCl_2(C_{21}H_{19}N_3)$ molecule (Scheme I, Fig. 1) lies on a twofold rotation axis that passes through the $N_{pyridyl}$—Cu bond; this symmetry element relates one half of the organic ligand to the other. The three N atoms span the axial–equatorial-axial sites of the trigonal bipyramidal coordination polyhedron; the geometry of Cu is 31% distorted along the Berry pseudorotation pathway.

S2. Experimental

2,6-Bis(*p*-tolylimino)pyridine (0.016 g, 0.05 mmol), and copper chloride dihydrate (0.01 g, 0.05 mmol) along with five drops of 1 *M* hydrochloric acid were dissolved in ethanol (10 ml). The mixture was heated in a Teflon-lined, stainless-steel Parr bomb at 363 K for 120 h. The bomb was cooled at 5 K per hour. Deep orange crystals were isolated.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.98 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 to 1.5*U*(C).

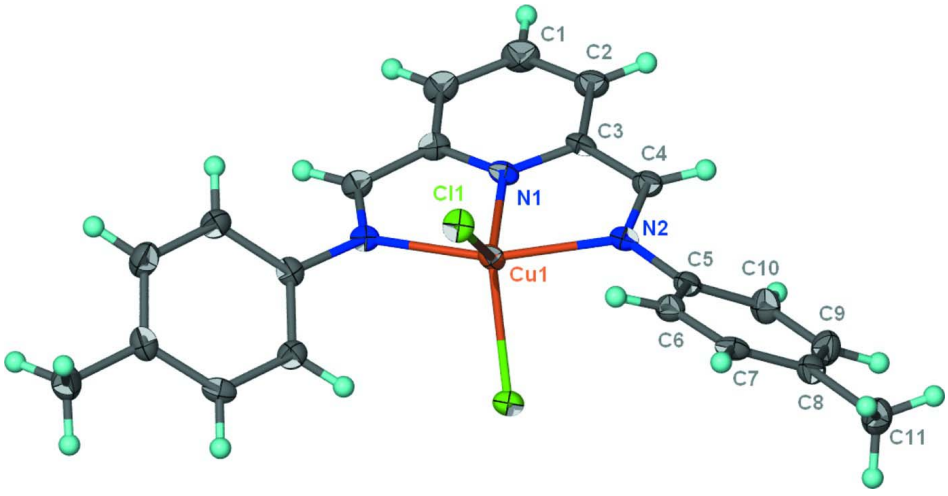

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $CuCl_2(C_{21}H_{19}N_3)$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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Crystal data

$[CuCl_2(C_{21}H_{19}N_3)]$
$M_r = 447.83$
Orthorhombic, *Fdd*2
Hall symbol: F 2 -2d
$a = 11.5220\ (13)$ Å
$b = 35.522\ (4)$ Å
$c = 9.327\ (1)$ Å
$V = 3817.4\ (7)$ Å^3
$Z = 8$
$F(000) = 1832$
$D_x = 1.558$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2394 reflections
$\theta = 2.3–26.1°$
$\mu = 1.44$ mm^{-1}
$T = 100$ K
Prism, orange
0.36 × 0.12 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.626$, $T_{max} = 0.972$
8753 measured reflections
2190 independent reflections
2023 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.3°$
$h = -13\rightarrow14$
$k = -46\rightarrow46$
$l = -12\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.070$
$S = 1.04$
2190 reflections
125 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0349P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.30$ e Å^{-3}
Absolute structure: Flack (1983), 858 Friedel pairs
Absolute structure parameter: 0.014 (14)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	1.0000	0.5000	0.50991 (4)	0.01349 (12)
Cl1	0.90550 (6)	0.543930 (17)	0.36785 (8)	0.01783 (15)
N1	1.0000	0.5000	0.7209 (3)	0.0136 (7)
N2	0.8670 (2)	0.46146 (6)	0.5570 (2)	0.0137 (5)
C1	1.0000	0.5000	1.0139 (7)	0.0233 (8)
H1	1.0000	0.5000	1.1158	0.028*
C2	0.9203 (3)	0.47824 (8)	0.9392 (3)	0.0197 (6)
H2	0.8648	0.4634	0.9889	0.024*
C3	0.9231 (2)	0.47860 (7)	0.7896 (3)	0.0148 (6)
C4	0.8502 (3)	0.45700 (8)	0.6925 (3)	0.0159 (6)
H4	0.7924	0.4403	0.7276	0.019*
C5	0.8043 (2)	0.43861 (7)	0.4590 (3)	0.0146 (5)

C6	0.7745 (2)	0.45364 (7)	0.3259 (3)	0.0162 (6)
H6	0.7996	0.4782	0.3003	0.019*
C7	0.7085 (2)	0.43273 (7)	0.2314 (3)	0.0155 (6)
H7	0.6854	0.4436	0.1429	0.019*
C8	0.6752 (2)	0.39602 (7)	0.2636 (3)	0.0184 (6)
C9	0.7099 (3)	0.38078 (8)	0.3943 (3)	0.0220 (6)
H9	0.6893	0.3556	0.4171	0.026*
C10	0.7736 (2)	0.40150 (8)	0.4910 (3)	0.0195 (6)
H10	0.7966	0.3906	0.5796	0.023*
C11	0.6078 (3)	0.37314 (8)	0.1571 (3)	0.0233 (6)
H11A	0.5523	0.3571	0.2079	0.035*
H11B	0.6613	0.3574	0.1017	0.035*
H11C	0.5659	0.3900	0.0921	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0124 (2)	0.0191 (2)	0.0089 (2)	−0.00124 (19)	0.000	0.000
Cl1	0.0175 (3)	0.0185 (3)	0.0175 (3)	0.0010 (3)	−0.0038 (3)	0.0027 (3)
N1	0.0098 (15)	0.0178 (15)	0.0131 (17)	0.0039 (13)	0.000	0.000
N2	0.0135 (12)	0.0152 (11)	0.0124 (11)	0.0023 (9)	0.0014 (9)	0.0001 (8)
C1	0.031 (2)	0.0271 (18)	0.0118 (18)	0.000 (2)	0.000	0.000
C2	0.0255 (16)	0.0207 (15)	0.0129 (14)	0.0006 (11)	0.0038 (11)	−0.0004 (11)
C3	0.0139 (14)	0.0179 (13)	0.0124 (16)	0.0031 (10)	0.0043 (11)	0.0007 (10)
C4	0.0180 (15)	0.0165 (13)	0.0133 (14)	0.0010 (11)	0.0023 (11)	0.0002 (11)
C5	0.0134 (13)	0.0177 (13)	0.0126 (13)	−0.0007 (11)	0.0018 (11)	−0.0017 (10)
C6	0.0166 (13)	0.0152 (12)	0.0168 (14)	0.0013 (11)	0.0016 (11)	0.0001 (11)
C7	0.0158 (13)	0.0211 (13)	0.0095 (14)	0.0048 (11)	0.0007 (10)	−0.0007 (10)
C8	0.0153 (13)	0.0215 (13)	0.0183 (14)	−0.0026 (10)	0.0005 (14)	−0.0032 (13)
C9	0.0271 (15)	0.0187 (14)	0.0201 (16)	−0.0057 (11)	0.0014 (13)	0.0012 (11)
C10	0.0212 (15)	0.0196 (13)	0.0177 (16)	−0.0026 (10)	−0.0009 (12)	0.0043 (12)
C11	0.0252 (16)	0.0243 (15)	0.0203 (16)	−0.0069 (13)	−0.0020 (12)	−0.0011 (12)

Geometric parameters (Å, º)

Cu1—N1	1.968 (3)	C4—H4	0.9500
Cu1—N2^{i}	2.101 (2)	C5—C6	1.394 (4)
Cu1—N2	2.101 (2)	C5—C10	1.397 (4)
Cu1—Cl1	2.3187 (7)	C6—C7	1.381 (4)
Cu1—Cl1^{i}	2.3187 (7)	C6—H6	0.9500
N1—C3^{i}	1.332 (3)	C7—C8	1.392 (4)
N1—C3	1.332 (3)	C7—H7	0.9500
N2—C4	1.288 (3)	C8—C9	1.392 (4)
N2—C5	1.421 (4)	C8—C11	1.500 (4)
C1—C2^{i}	1.388 (5)	C9—C10	1.377 (4)
C1—C2	1.388 (5)	C9—H9	0.9500
C1—H1	0.9500	C10—H10	0.9500
C2—C3	1.396 (3)	C11—H11A	0.9800

C2—H2	0.9500	C11—H11B	0.9800
C3—C4	1.454 (4)	C11—H11C	0.9800
N1—Cu1—N2^{i}	77.92 (7)	N2—C4—H4	121.3
N1—Cu1—N2	77.92 (7)	C3—C4—H4	121.3
N2^{i}—Cu1—N2	155.85 (13)	C6—C5—C10	119.3 (3)
N1—Cu1—Cl1	124.85 (2)	C6—C5—N2	118.7 (2)
N2^{i}—Cu1—Cl1	91.35 (6)	C10—C5—N2	122.0 (2)
N2—Cu1—Cl1	102.45 (7)	C7—C6—C5	119.8 (2)
N1—Cu1—Cl1^{i}	124.85 (2)	C7—C6—H6	120.1
N2^{i}—Cu1—Cl1^{i}	102.45 (7)	C5—C6—H6	120.1
N2—Cu1—Cl1^{i}	91.35 (6)	C6—C7—C8	121.2 (3)
Cl1—Cu1—Cl1^{i}	110.30 (4)	C6—C7—H7	119.4
C3^{i}—N1—C3	122.4 (3)	C8—C7—H7	119.4
C3^{i}—N1—Cu1	118.78 (17)	C7—C8—C9	118.3 (3)
C3—N1—Cu1	118.78 (17)	C7—C8—C11	120.5 (3)
C4—N2—C5	119.0 (2)	C9—C8—C11	121.2 (2)
C4—N2—Cu1	113.3 (2)	C10—C9—C8	121.3 (3)
C5—N2—Cu1	127.49 (18)	C10—C9—H9	119.4
C2^{i}—C1—C2	119.7 (5)	C8—C9—H9	119.4
C2^{i}—C1—H1	120.1	C9—C10—C5	119.9 (3)
C2—C1—H1	120.1	C9—C10—H10	120.0
C1—C2—C3	118.8 (4)	C5—C10—H10	120.0
C1—C2—H2	120.6	C8—C11—H11A	109.5
C3—C2—H2	120.6	C8—C11—H11B	109.5
N1—C3—C2	120.2 (3)	H11A—C11—H11B	109.5
N1—C3—C4	112.7 (2)	C8—C11—H11C	109.5
C2—C3—C4	127.2 (3)	H11A—C11—H11C	109.5
N2—C4—C3	117.3 (3)	H11B—C11—H11C	109.5
N2^{i}—Cu1—N1—C3^{i}	−1.14 (14)	C1—C2—C3—N1	−1.1 (4)
N2—Cu1—N1—C3^{i}	178.86 (14)	C1—C2—C3—C4	177.8 (2)
Cl1—Cu1—N1—C3^{i}	−84.27 (13)	C5—N2—C4—C3	174.8 (2)
Cl1^{i}—Cu1—N1—C3^{i}	95.73 (13)	Cu1—N2—C4—C3	0.1 (3)
N2^{i}—Cu1—N1—C3	178.86 (14)	N1—C3—C4—N2	−1.0 (4)
N2—Cu1—N1—C3	−1.14 (14)	C2—C3—C4—N2	−180.0 (3)
Cl1—Cu1—N1—C3	95.73 (13)	C4—N2—C5—C6	148.0 (3)
Cl1^{i}—Cu1—N1—C3	−84.27 (13)	Cu1—N2—C5—C6	−38.1 (3)
N1—Cu1—N2—C4	0.5 (2)	C4—N2—C5—C10	−33.1 (4)
N2^{i}—Cu1—N2—C4	0.5 (2)	Cu1—N2—C5—C10	140.8 (2)
Cl1—Cu1—N2—C4	−122.9 (2)	C10—C5—C6—C7	4.4 (4)
Cl1^{i}—Cu1—N2—C4	126.0 (2)	N2—C5—C6—C7	−176.7 (2)
N1—Cu1—N2—C5	−173.7 (2)	C5—C6—C7—C8	−3.0 (4)
N2^{i}—Cu1—N2—C5	−173.7 (2)	C6—C7—C8—C9	0.2 (4)
Cl1—Cu1—N2—C5	62.9 (2)	C6—C7—C8—C11	−177.4 (3)
Cl1^{i}—Cu1—N2—C5	−48.3 (2)	C7—C8—C9—C10	1.3 (4)
C2^{i}—C1—C2—C3	0.5 (2)	C11—C8—C9—C10	178.8 (3)
C3^{i}—N1—C3—C2	0.6 (2)	C8—C9—C10—C5	0.1 (4)

Cu1—N1—C3—C2	−179.4 (2)	C6—C5—C10—C9	−3.0 (4)
C3^{i}—N1—C3—C4	−178.5 (2)	N2—C5—C10—C9	178.2 (3)
Cu1—N1—C3—C4	1.5 (2)		

Symmetry code: (i) −*x*+2, −*y*+1, *z*.