Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Xiao-Ping Li,[a] Jian-She Zhao[a] and Seik Weng Ng[b]*

[a]Department of Chemistry, Shaanxi Key Laboratory for Physico-Inorganic Chemistry, Northwest University, Xi'an 710069, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

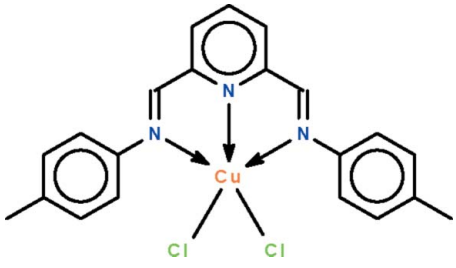
Received 15 September 2010; accepted 16 September 2010

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).

We thank the Graduate Experimental Research Fund of Northwest University (project No. 09YSY22), the National Natural Science Foundation of China (No. 20971104) and the University of Malaya for supporting this study.

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

References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Li, X.-P., Liu, Y.-Y. & Zhao, J.-S. (2010). *Acta Cryst.* E**66**, m1215.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

Acta Cryst. (2010). E**66**, m1297 [**doi:10.1107/S1600536810037025**]

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

X.-P. Li, J.-S. Zhao and S. W. Ng

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.

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.

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).

Figures

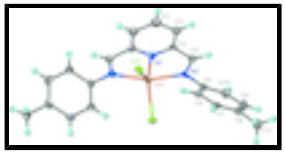


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $CuCl_2(C_{21}H_{19}N_3)$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$[CuCl_2(C_{21}H_{19}N_3)]$	$F(000) = 1832$
$M_r = 447.83$	$D_x = 1.558$ Mg m^{-3}
Orthorhombic, *Fdd*2	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: F 2 -2d	Cell parameters from 2394 reflections
$a = 11.5220$ (13) Å	θ = 2.3–26.1°
$b = 35.522$ (4) Å	μ = 1.44 mm^{-1}
$c = 9.327$ (1) Å	$T = 100$ K

V = 3817.4 (7) Å^3	Prism, orange
Z = 8	0.36 × 0.12 × 0.02 mm

Data collection

Bruker SMART APEX diffractometer	2190 independent reflections
Radiation source: fine-focus sealed tube	2023 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.050
ω scans	θ_{max} = 27.5°, θ_{min} = 2.3°
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	h = −13→14
T_{min} = 0.626, T_{max} = 0.972	k = −46→46
8753 measured reflections	l = −12→12

Refinement

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

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

	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 (Å^2)

	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, °)

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 codes: (i) −*x*+2, −*y*+1, *z*.

Fig. 1