metal-organic compounds

Dichlorido{N,N-dimethyl-N'-[1-(2-pyridyl)ethylidene]ethane-1,2-diamine-κ³N,N',N''}copper(II)

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In the title compound, [CuCl₂(C₁₁H₁₇N₃)], the Cuᴵᴵ ion is five-coordinated with a distorted square-pyramidal configuration. The three N atoms of the Schiff base ligand and one Cl atom are located in the basal plane, whereas the other Cl atom is apically positioned.

Related literature

For the crystal structures of similar copper (II) complexes, see: Wang *et al.* (2009); Yuan & Zhang (2005); Zhang *et al.* (2009). For a description of the geometry of five-coordinated metal complexes, see: Addison *et al.* (1984).
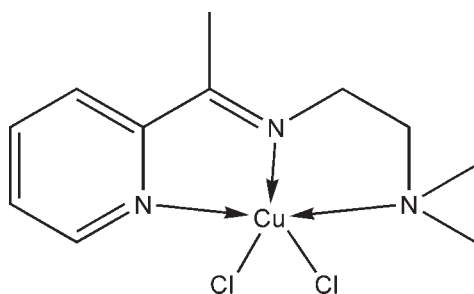
Experimental

Crystal data

[CuCl₂(C₁₁H₁₇N₃)]	V = 1381.51 (2) Å³
M_r = 325.72	Z = 4
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
a = 9.81448 (12) Å	μ = 1.95 mm⁻¹
b = 9.90297 (13) Å	T = 100 K
c = 14.21414 (18) Å	0.30 × 0.23 × 0.07 mm

Data collection

Bruker APEXII CCD diffractometer	10998 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	2439 independent reflections
	2378 reflections with $I > 2\sigma(I)$
T_{min} = 0.592, T_{max} = 0.876	R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.017	$\Delta\rho_{max}$ = 0.21 e Å⁻³
$wR(F^2)$ = 0.043	$\Delta\rho_{min}$ = −0.20 e Å⁻³
S = 1.03	Absolute structure: Flack (1983),
2439 reflections	1022 Friedel pairs
157 parameters	Flack parameter: 0.010 (9)
H-atom parameters constrained	

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); 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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supporting information

Dichlorido{*N,N*-dimethyl-*N′*-[1-(2-pyridyl)ethylidene]ethane-1,2-diamine-$\kappa^3 N,N',N''$}copper(II)

Muhammad Saleh Salga, Hamid Khaledi, Hapipah Mohd Ali and Rustam Puteh

S1. Comment

The title compound was obtained by the reaction of *N,N*-dimethyl-*N′*-[methyl(2-pyridyl)methylene]ethane-1,2-diamine with copper(II) chloride. In the molecule of the complex, the metal ion is penta-coordinated by the tridentate Schiff base ligand and two chloride atoms (Fig. 1). The geometry of the complex can be determined by using the index $\tau = (\beta-\alpha)/60$, where β is the largest angle and α is the second one around the metal center. For an ideal square-pyramidal geometry τ is 0, while it is 1 in a perfect trigonal-bipyramid (Addison *et al.*,1984). The two largest angels in the title compound are 158.45 (6)° (N1—Cu—N3) and 154.98 (5)° (N2—Cu—Cl2) which give a τ value of 0.058. This value indicates a slightly distorted square pyramidal geometry in which the three N atoms of the Schiff base ligand and one chloride atom occupy the basal positions and the other chloride atom is placed in the apical position.

S2. Experimental

The Schiff base ligand was prepared *via* condensation reaction of *N,N*-dimethylethyldiamine (0.44 g, 5 mmol) and 2-acetylpyridine (0.61 g, 5 mmol) by refluxing in ethanol (50 ml) for 2 h. For synthesis of the title complex a mixture of the Schiff base ligand (0.57 g, 3 mmol) and copper (II) chloride dihydrate (0.51 g, 3 mmol) in ethanol (50 ml) was stirred at room temperature for half an hour. The solvent was then evaporated partially to yield the title complex as a green solid. Suitable crystals for X-ray crystallography were obtained upon slow evaporation of an ethanolic solution at room temperature.

S3. Refinement

Hydrogen atoms were placed at calculated positions (C—H 0.95-0.98 Å), and were treated as riding on their parent atoms, with U_{iso}(H) set to 1.2-1.5 times U_{eq}(C). An absolute structure was established using anomalous dispersion effects; 1021 Friedel pairs were not merged.
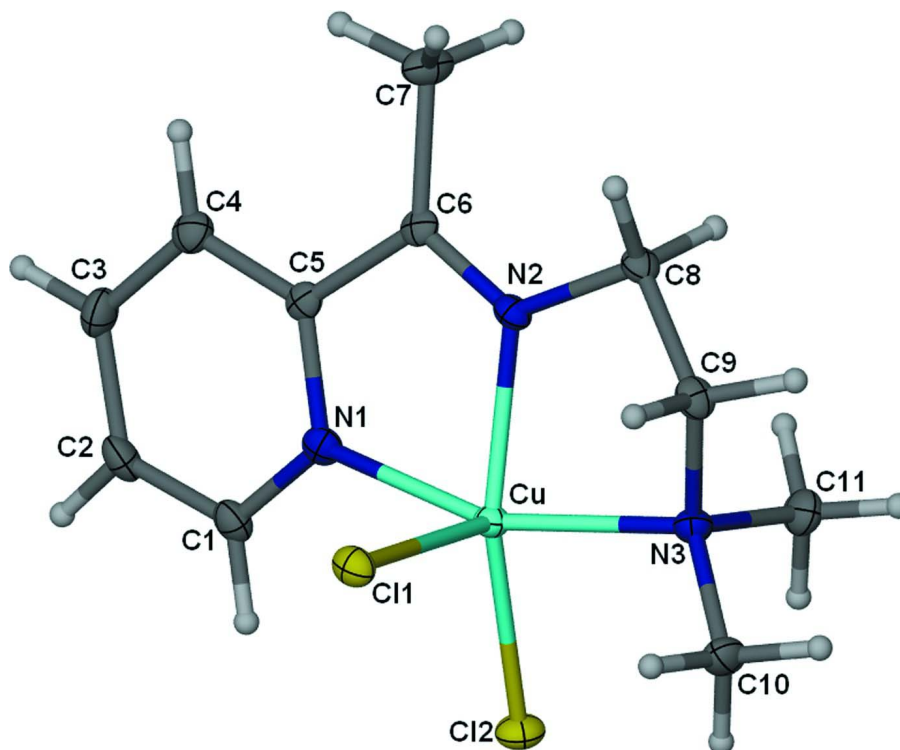
Figure 1

Thermal ellipsoid plot of the title compound at the 50% probability level.

Dichlorido{*N*,*N*-dimethyl-*N′*-[1-(2- pyridyl)ethylidene]ethane-1,2-diamine- *κ³N,N′,N″*}copper(II)

Crystal data

$[CuCl_2(C_{11}H_{17}N_3)]$	$F(000) = 668$
$M_r = 325.72$	$D_x = 1.566$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 7155 reflections
$a = 9.81448$ (12) Å	$\theta = 2.5$–30.4°
$b = 9.90297$ (13) Å	$\mu = 1.95$ mm^{-1}
$c = 14.21414$ (18) Å	$T = 100$ K
$V = 1381.51$ (2) Å^3	Block, green
$Z = 4$	$0.30 \times 0.23 \times 0.07$ mm

Data collection

Bruker APEXII CCD diffractometer	10998 measured reflections
Radiation source: fine-focus sealed tube	2439 independent reflections
Graphite monochromator	2378 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.026$
Absorption correction: multi-scan	$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$
(*SADABS*; Sheldrick, 1996)	$h = -11 \rightarrow 11$
$T_{min} = 0.592$, $T_{max} = 0.876$	$k = -11 \rightarrow 11$
	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.017$	H-atom parameters constrained
$wR(F^2) = 0.043$	$w = 1/[\sigma^2(F_o^2) + (0.0245P)^2]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
2439 reflections	$(\Delta/\sigma)_{max} = 0.001$
157 parameters	$\Delta\rho_{max} = 0.21$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.20$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1022 Friedel pairs
Secondary atom site location: difference Fourier map	Absolute structure parameter: 0.010 (9)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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	$U_{iso}*/U_{eq}$
Cu	0.80028 (2)	0.16329 (2)	0.099598 (15)	0.01175 (7)
Cl1	0.56577 (5)	0.08033 (5)	0.13120 (3)	0.01619 (11)
Cl2	0.92633 (5)	0.14346 (5)	0.23208 (3)	0.01790 (12)
N1	0.86920 (16)	−0.00757 (17)	0.03434 (12)	0.0139 (4)
N2	0.76828 (16)	0.20963 (16)	−0.03353 (11)	0.0124 (4)
N3	0.75938 (15)	0.36500 (16)	0.11931 (11)	0.0132 (4)
C1	0.9300 (2)	−0.11396 (19)	0.07379 (15)	0.0169 (4)
H1	0.9448	−0.1136	0.1398	0.020*
C2	0.9722 (2)	−0.2245 (2)	0.02225 (15)	0.0185 (5)
H2	1.0174	−0.2976	0.0522	0.022*
C3	0.9479 (2)	−0.2274 (2)	−0.07327 (15)	0.0200 (5)
H3	0.9737	−0.3036	−0.1098	0.024*
C4	0.8846 (2)	−0.1161 (2)	−0.11553 (15)	0.0175 (4)
H4	0.8666	−0.1155	−0.1812	0.021*
C5	0.84897 (19)	−0.0075 (2)	−0.06026 (13)	0.0131 (4)
C6	0.79321 (18)	0.12257 (18)	−0.09696 (13)	0.0129 (4)
C7	0.7771 (2)	0.1485 (2)	−0.20048 (13)	0.0200 (5)
H7A	0.6843	0.1805	−0.2132	0.030*
H7B	0.7935	0.0648	−0.2354	0.030*
H7C	0.8428	0.2173	−0.2204	0.030*
C8	0.71958 (19)	0.3470 (2)	−0.05216 (13)	0.0139 (4)
H8A	0.6464	0.3460	−0.1001	0.017*
H8B	0.7949	0.4047	−0.0751	0.017*

C9	0.6657 (2)	0.3992 (2)	0.04119 (14)	0.0158 (4)
H9A	0.6548	0.4985	0.0377	0.019*
H9B	0.5751	0.3592	0.0536	0.019*
C10	0.6923 (2)	0.3973 (2)	0.20995 (13)	0.0195 (4)
H10A	0.7533	0.3737	0.2620	0.029*
H10B	0.6076	0.3456	0.2154	0.029*
H10C	0.6718	0.4941	0.2124	0.029*
C11	0.8874 (2)	0.4429 (2)	0.11176 (15)	0.0200 (5)
H11A	0.9314	0.4230	0.0514	0.030*
H11B	0.9486	0.4176	0.1633	0.030*
H11C	0.8671	0.5396	0.1155	0.030*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu	0.01269 (12)	0.01237 (12)	0.01020 (12)	0.00077 (10)	−0.00026 (10)	0.00045 (10)
Cl1	0.0134 (2)	0.0185 (3)	0.0166 (2)	−0.0024 (2)	−0.00053 (18)	0.0027 (2)
Cl2	0.0177 (2)	0.0229 (3)	0.0130 (2)	0.0021 (2)	−0.00368 (19)	0.0008 (2)
N1	0.0115 (8)	0.0159 (9)	0.0143 (8)	−0.0012 (7)	0.0026 (7)	0.0014 (7)
N2	0.0107 (9)	0.0133 (8)	0.0131 (8)	0.0003 (6)	−0.0012 (7)	0.0033 (7)
N3	0.0137 (8)	0.0146 (8)	0.0114 (8)	0.0001 (6)	0.0009 (6)	0.0000 (7)
C1	0.0181 (10)	0.0146 (10)	0.0180 (11)	−0.0018 (8)	0.0028 (9)	0.0036 (8)
C2	0.0203 (11)	0.0136 (11)	0.0217 (12)	0.0004 (8)	0.0034 (9)	0.0057 (9)
C3	0.0229 (11)	0.0144 (10)	0.0227 (11)	−0.0009 (9)	0.0024 (9)	−0.0051 (9)
C4	0.0170 (10)	0.0184 (11)	0.0171 (11)	−0.0034 (8)	−0.0009 (9)	−0.0014 (9)
C5	0.0101 (10)	0.0148 (10)	0.0143 (10)	−0.0034 (8)	0.0011 (8)	−0.0012 (8)
C6	0.0093 (9)	0.0160 (9)	0.0134 (9)	−0.0037 (7)	−0.0012 (9)	−0.0009 (8)
C7	0.0223 (11)	0.0237 (11)	0.0139 (10)	0.0061 (10)	−0.0012 (8)	−0.0014 (9)
C8	0.0145 (10)	0.0136 (10)	0.0137 (10)	0.0008 (9)	−0.0019 (7)	0.0020 (8)
C9	0.0146 (10)	0.0144 (10)	0.0185 (10)	0.0017 (8)	−0.0009 (8)	0.0019 (9)
C10	0.0272 (11)	0.0176 (10)	0.0136 (10)	0.0018 (10)	0.0029 (10)	−0.0021 (8)
C11	0.0213 (11)	0.0183 (11)	0.0204 (12)	−0.0064 (8)	−0.0018 (9)	−0.0015 (10)

Geometric parameters (Å, º)

Cu—N2	1.9723 (16)	C4—C5	1.377 (3)
Cu—N1	2.0447 (17)	C4—H4	0.9500
Cu—N3	2.0567 (16)	C5—C6	1.494 (3)
Cu—Cl2	2.2617 (5)	C6—C7	1.502 (3)
Cu—Cl1	2.4848 (5)	C7—H7A	0.9800
N1—C1	1.334 (3)	C7—H7B	0.9800
N1—C5	1.359 (2)	C7—H7C	0.9800
N2—C6	1.271 (2)	C8—C9	1.519 (3)
N2—C8	1.466 (3)	C8—H8A	0.9900
N3—C11	1.478 (2)	C8—H8B	0.9900
N3—C10	1.482 (2)	C9—H9A	0.9900
N3—C9	1.481 (2)	C9—H9B	0.9900
C1—C2	1.381 (3)	C10—H10A	0.9800

C1—H1	0.9500	C10—H10B	0.9800
C2—C3	1.379 (3)	C10—H10C	0.9800
C2—H2	0.9500	C11—H11A	0.9800
C3—C4	1.401 (3)	C11—H11B	0.9800
C3—H3	0.9500	C11—H11C	0.9800
N2—Cu—N1	79.04 (7)	N1—C5—C6	113.54 (17)
N2—Cu—N3	82.74 (6)	C4—C5—C6	124.56 (17)
N1—Cu—N3	158.45 (6)	N2—C6—C5	114.06 (16)
N2—Cu—Cl2	154.98 (5)	N2—C6—C7	123.94 (18)
N1—Cu—Cl2	97.17 (5)	C5—C6—C7	121.90 (16)
N3—Cu—Cl2	94.45 (4)	C6—C7—H7A	109.5
N2—Cu—Cl1	95.91 (5)	C6—C7—H7B	109.5
N1—Cu—Cl1	96.59 (5)	H7A—C7—H7B	109.5
N3—Cu—Cl1	96.65 (4)	C6—C7—H7C	109.5
Cl2—Cu—Cl1	109.113 (18)	H7A—C7—H7C	109.5
C1—N1—C5	118.77 (18)	H7B—C7—H7C	109.5
C1—N1—Cu	127.64 (14)	N2—C8—C9	105.76 (15)
C5—N1—Cu	113.59 (13)	N2—C8—H8A	110.6
C6—N2—C8	124.33 (16)	C9—C8—H8A	110.6
C6—N2—Cu	119.47 (13)	N2—C8—H8B	110.6
C8—N2—Cu	116.17 (12)	C9—C8—H8B	110.6
C11—N3—C10	109.14 (15)	H8A—C8—H8B	108.7
C11—N3—C9	110.72 (15)	N3—C9—C8	111.17 (16)
C10—N3—C9	109.07 (15)	N3—C9—H9A	109.4
C11—N3—Cu	109.33 (12)	C8—C9—H9A	109.4
C10—N3—Cu	114.53 (12)	N3—C9—H9B	109.4
C9—N3—Cu	103.96 (12)	C8—C9—H9B	109.4
N1—C1—C2	122.50 (19)	H9A—C9—H9B	108.0
N1—C1—H1	118.8	N3—C10—H10A	109.5
C2—C1—H1	118.8	N3—C10—H10B	109.5
C3—C2—C1	119.1 (2)	H10A—C10—H10B	109.5
C3—C2—H2	120.4	N3—C10—H10C	109.5
C1—C2—H2	120.4	H10A—C10—H10C	109.5
C2—C3—C4	118.9 (2)	H10B—C10—H10C	109.5
C2—C3—H3	120.6	N3—C11—H11A	109.5
C4—C3—H3	120.6	N3—C11—H11B	109.5
C5—C4—C3	118.86 (19)	H11A—C11—H11B	109.5
C5—C4—H4	120.6	N3—C11—H11C	109.5
C3—C4—H4	120.6	H11A—C11—H11C	109.5
N1—C5—C4	121.81 (19)	H11B—C11—H11C	109.5