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[1,2-Bis(pyridin-2-ylmethoxy)benzene- κ^4N,O,O',N']dichloridocopper(II)Nan-Nan Huang,^a Shuang Zhang,^b Ying Liu,^c Guang-Feng Hou^b and Jin-Sheng Gao^{b,*}

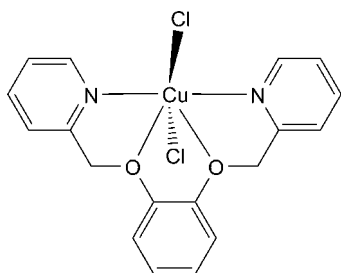
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.034; wR factor = 0.081; data-to-parameter ratio = 17.9.

In the title compound, $[\text{CuCl}_2(\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_2)]$, the Cu^{II} atom lies on a twofold axis and is six-coordinated in a distorted octahedral environment defined by two N and two O atoms from the ligand and by two Cl atoms. In the crystal, $\pi-\pi$ interactions [centroid-centroid distance = $3.838(1)$ Å] and $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds link adjacent molecules into a chain structure along $[101]$.

Related literature

For related structures, see: Zhang *et al.* (2010a,b).

Experimental

Crystal data

 $[\text{CuCl}_2(\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_2)]$ $M_r = 426.77$

Monoclinic, $C2/c$
 $a = 10.624(2)$ Å
 $b = 19.458(4)$ Å
 $c = 8.8063(18)$ Å
 $\beta = 101.35(3)^\circ$
 $V = 1784.8(6)$ Å³

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 1.54$ mm⁻¹
 $T = 293$ K
 $0.21 \times 0.19 \times 0.16$ mm

Data collection

Rigaku R-Axis RAPID
diffractometer
Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.739$, $T_{\max} = 0.790$

7741 measured reflections
2045 independent reflections
1637 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.081$
 $S = 1.05$
2045 reflections

114 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.31$ e Å⁻³
 $\Delta\rho_{\min} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C6}-\text{H6A}\cdots\text{Cl1}^i$	0.97	2.65	3.541 (3)	153

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5145).

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supporting information

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[1,2-Bis(pyridin-2-ylmethoxy)benzene- κ^4 N,O,O',N']dichloridocopper(II)

Nan-Nan Huang, Shuang Zhang, Ying Liu, Guang-Feng Hou and Jin-Sheng Gao

S1. Comment

N-heterocyclic ligands coordinated with transition metal ions can form a variety of topology structures, including macrocycles, polyhedra and linear and helical polymers. Our group has report three kinds of flexible pyridyl-based ligands in the previous report. As a part of our continuing work for bipyridyl aromatic ligands, we report the crystal structure of the title compound here.

1,2-Bis(pyridin-2-ylmethoxy)benzene molecule act as a chelating ligand to coordinate with Cu^{II} ion forming a discrete strucutre. Two chlorid counter ions also coordinate to the center Cu^{II} ion, resulting a sxi-coordinated distorted octahedral geometry environment (Figure 1).

In the crystal, the π — π interactions with distance about 3.838 (1) Å, and the C—H $\cdots$ Cl hydrogen bonds link these isolated molecules to form a chain structure along [101] direction (Figure 2, Table 1).

S2. Experimental

The 1,2-Bis(pyridin-2-ylmethoxy)benzene was synthesized by the reaction of *o*-dihydroxybenzene and 2-chloromethylpyridine hydrochloride under nitrogen atmosphere and alkaline condition (Zhang *et al.*, 2010*a*). Title ligand (0.58 g, 0.02 mol) and CuCl₂ (0.27 g, 0.02 mol) were dissolved in 15 mL e thanol, and then the mixture keep stirring for 30 minute. The resulting solution was filtered, and the filtrate was allowed to stand in a desiccator at room temperature for several days. Bule needle crystals were obtained with yield 57%.

S3. Refinement

H atoms bound to C atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93 Å (aromatic C), C—H = 0.97 Å (methene C), and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

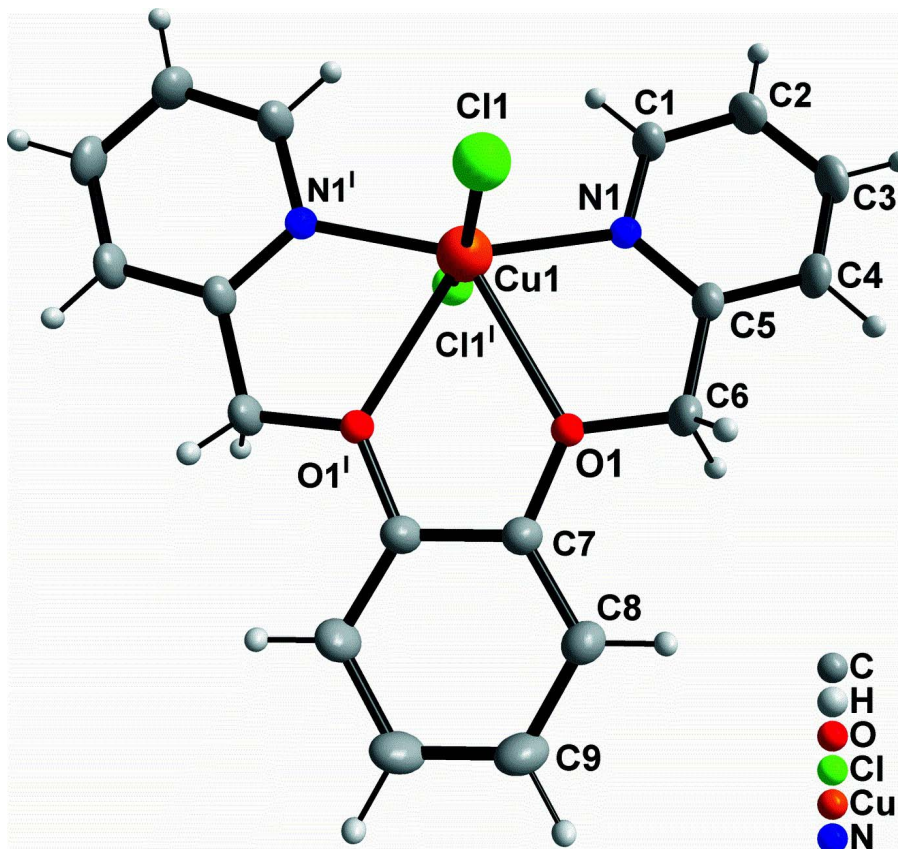


Figure 1

The molecular structure of the title compound, showing displacement ellipsoids at the 50% probability level for non-H atoms. Symmetry code I: $1 - x, y, 1.5 - z$.

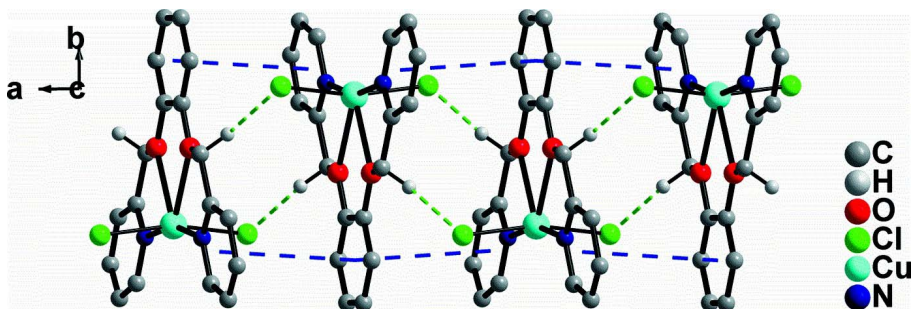


Figure 2

A partial packing view, showing the chain structure. Dashed lines indicate the hydrogen bonds (green) and π — π interactions (blue), no involving H atoms have been omitted for clarity.

[1,2-Bis(pyridin-2-ylmethoxy)benzene- κ^4N,O,O',N']dichloridocopper(II)

Crystal data

$[\text{CuCl}_2(\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_2)]$

$M_r = 426.77$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 10.624\ (2)\ \text{\AA}$

$b = 19.458\ (4)\ \text{\AA}$

$c = 8.8063\ (18)\ \text{\AA}$

$\beta = 101.35\ (3)^\circ$

$V = 1784.8 (6) \text{ \AA}^3$
 $Z = 4$
 $F(000) = 868$
 $D_x = 1.588 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 6110 reflections

$\theta = 3.2\text{--}27.5^\circ$
 $\mu = 1.54 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Block, green
 $0.21 \times 0.19 \times 0.16 \text{ mm}$

Data collection

Rigaku R-AXIS RAPID
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.739$, $T_{\max} = 0.790$

7741 measured reflections
 2045 independent reflections
 1637 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$
 $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.2^\circ$
 $h = -13 \rightarrow 13$
 $k = -25 \rightarrow 25$
 $l = -11 \rightarrow 9$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.081$
 $S = 1.05$
 2045 reflections
 114 parameters
 0 restraints
 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
 $w = 1/[\sigma^2(F_o^2) + (0.0321P)^2 + 1.5862P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.31 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e \AA}^{-3}$

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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3928 (2)	0.42692 (13)	0.4854 (3)	0.0455 (6)
H1	0.4104	0.4597	0.5635	0.055*
C2	0.3430 (2)	0.44885 (14)	0.3369 (3)	0.0474 (6)
H2	0.3282	0.4953	0.3152	0.057*
C3	0.3159 (2)	0.40034 (14)	0.2217 (3)	0.0480 (6)
H3	0.2825	0.4135	0.1203	0.058*
C4	0.3387 (2)	0.33231 (13)	0.2581 (2)	0.0431 (6)
H4	0.3195	0.2988	0.1817	0.052*
C5	0.3910 (2)	0.31374 (12)	0.4105 (2)	0.0363 (5)
C6	0.4195 (3)	0.24013 (13)	0.4492 (3)	0.0471 (6)
H6A	0.3451	0.2122	0.4074	0.057*

H6B	0.4904	0.2249	0.4031	0.057*
O1	0.4510 (2)	0.23204 (9)	0.60859 (18)	0.0680 (6)
C7	0.4747 (2)	0.16783 (12)	0.6701 (3)	0.0445 (6)
C8	0.4510 (2)	0.10685 (13)	0.5912 (3)	0.0489 (6)
H8	0.4186	0.1067	0.4851	0.059*
C9	0.4759 (3)	0.04561 (14)	0.6716 (3)	0.0555 (7)
H9	0.4600	0.0041	0.6191	0.067*
Cl1	0.69527 (6)	0.35669 (4)	0.68252 (7)	0.0570 (2)
Cu1	0.5000	0.34350 (2)	0.7500	0.03802 (14)
N1	0.41728 (18)	0.36093 (10)	0.52330 (19)	0.0375 (4)

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0599 (16)	0.0429 (14)	0.0324 (12)	−0.0005 (11)	0.0060 (10)	0.0007 (10)
C2	0.0516 (15)	0.0499 (15)	0.0402 (13)	0.0055 (12)	0.0078 (10)	0.0116 (11)
C3	0.0450 (14)	0.0692 (18)	0.0282 (12)	0.0031 (12)	0.0033 (9)	0.0103 (11)
C4	0.0443 (14)	0.0569 (16)	0.0265 (10)	−0.0026 (11)	0.0032 (9)	−0.0041 (10)
C5	0.0385 (12)	0.0432 (13)	0.0270 (11)	−0.0039 (10)	0.0059 (8)	−0.0025 (9)
C6	0.0662 (16)	0.0438 (14)	0.0292 (11)	−0.0031 (12)	0.0041 (10)	−0.0045 (10)
O1	0.1351 (19)	0.0334 (10)	0.0290 (9)	0.0025 (10)	0.0000 (9)	−0.0006 (7)
C7	0.0616 (16)	0.0345 (13)	0.0387 (12)	−0.0009 (11)	0.0133 (11)	−0.0010 (9)
C8	0.0631 (16)	0.0415 (14)	0.0444 (14)	−0.0022 (12)	0.0164 (11)	−0.0072 (11)
C9	0.0749 (19)	0.0355 (14)	0.0625 (15)	−0.0039 (12)	0.0287 (14)	−0.0074 (11)
Cl1	0.0554 (4)	0.0796 (5)	0.0357 (3)	0.0167 (3)	0.0081 (3)	0.0120 (3)
Cu1	0.0557 (3)	0.0337 (2)	0.02259 (19)	0.000	0.00265 (15)	0.000
N1	0.0463 (11)	0.0392 (11)	0.0254 (9)	−0.0022 (8)	0.0032 (7)	0.0012 (7)

Geometric parameters ($\text{\AA}$, $^\circ$)

C1—N1	1.339 (3)	C6—H6B	0.9700
C1—C2	1.379 (3)	O1—C7	1.365 (3)
C1—H1	0.9300	O1—Cu1	2.5040 (18)
C2—C3	1.374 (4)	C7—C8	1.373 (3)
C2—H2	0.9300	C7—C7 ⁱ	1.405 (5)
C3—C4	1.372 (4)	C8—C9	1.385 (4)
C3—H3	0.9300	C8—H8	0.9300
C4—C5	1.395 (3)	C9—C9 ⁱ	1.375 (5)
C4—H4	0.9300	C9—H9	0.9300
C5—N1	1.341 (3)	Cl1—Cu1	2.2820 (8)
C5—C6	1.490 (3)	Cu1—N1 ⁱ	2.0451 (18)
C6—O1	1.386 (3)	Cu1—N1	2.0451 (18)
C6—H6A	0.9700	Cu1—Cl1 ⁱ	2.2820 (8)
N1—C1—C2	123.5 (2)	C6—O1—Cu1	112.98 (14)
N1—C1—H1	118.2	O1—C7—C8	126.1 (2)
C2—C1—H1	118.2	O1—C7—C7 ⁱ	113.68 (12)
C3—C2—C1	118.2 (2)	C8—C7—C7 ⁱ	120.18 (15)

C3—C2—H2	120.9	C7—C8—C9	119.2 (2)
C1—C2—H2	120.9	C7—C8—H8	120.4
C4—C3—C2	119.3 (2)	C9—C8—H8	120.4
C4—C3—H3	120.4	C9 ⁱ —C9—C8	120.63 (15)
C2—C3—H3	120.4	C9 ⁱ —C9—H9	119.7
C3—C4—C5	119.5 (2)	C8—C9—H9	119.7
C3—C4—H4	120.2	N1 ⁱ —Cu1—N1	160.91 (11)
C5—C4—H4	120.2	N1 ⁱ —Cu1—Cl1	89.86 (6)
N1—C5—C4	121.4 (2)	N1—Cu1—Cl1	88.01 (6)
N1—C5—C6	119.02 (19)	N1 ⁱ —Cu1—Cl1 ⁱ	88.01 (6)
C4—C5—C6	119.6 (2)	N1—Cu1—Cl1 ⁱ	89.86 (6)
O1—C6—C5	109.80 (19)	Cl1—Cu1—Cl1 ⁱ	167.08 (4)
O1—C6—H6A	109.7	N1 ⁱ —Cu1—O1	129.53 (7)
C5—C6—H6A	109.7	N1—Cu1—O1	69.56 (7)
O1—C6—H6B	109.7	Cl1—Cu1—O1	94.51 (6)
C5—C6—H6B	109.7	Cl1 ⁱ —Cu1—O1	96.67 (6)
H6A—C6—H6B	108.2	C1—N1—C5	118.03 (19)
C7—O1—C6	119.62 (18)	C1—N1—Cu1	115.29 (15)
C7—O1—Cu1	126.26 (14)	C5—N1—Cu1	126.61 (16)

Symmetry code: (i) $-x+1, y, -z+3/2$.

Hydrogen-bond geometry ($\text{\AA}$, $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C6—H6A $\cdots$ Cl1 ⁱⁱ	0.97	2.65	3.541 (3)	153

Symmetry code: (ii) $x-1/2, -y+1/2, z-1/2$.