C37	0.348 (1)	0.434 (1)	0.1961 (8)	0.064 (5)
C38	0.330 (2)	0.543 (2)	0.219 (1)	0.083 (6)
C39	0.199 (2)	0.592 (1)	0.204 (1)	0.100 (6)
C40	0.071 (2)	0.529 (1)	0.1654 (8)	0.071 (5)

Table 2. *Selected geometric parameters* (Å, °)

Pd1—S1	2.323 (3)	P2—C23	1.83 (1)
Pd1—S2	2.300 (3)	P2—C29	1.83 (1)
Pd1—P1	2.299 (3)	P2—C35	1.83 (1)
Pd1—P2	2.332 (3)	N1—C3	1.13 (1)
S1—C1	1.73 (1)	N2—C4	1.11 (2)
S2—C2	1.73 (1)	C1—C2	1.35 (1)
P1—C5	1.820 (9)	C1—C3	1.44 (1)
P1—C11	1.82 (1)	C2—C4	1.45 (2)
P1—C17	1.83 (1)		
S1—Pd1—S2	88.75 (10)	S2—Pd1—P1	88.83 (10)
S1—Pd1—P1	170.3 (1)	S2—Pd1—P2	165.36 (9)
S1—Pd1—P2	87.31 (10)	P1—Pd1—P2	97.28 (9)

H atoms were fixed at calculated positions with $U_{\rm iso} = 1.2 \times U_{\rm eq}$ of the C atoms to which they are attached. The weighting scheme used included a 'fudge factor' p based on multiple measurements for the three standard reflections. Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1993). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN TABLES*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1118). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dichloro[6-methyl-2-(2-pyridylmethyl)pyridine-*N*,*N*′]copper(II)

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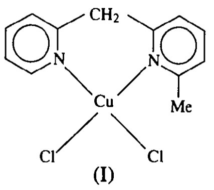
Abstract

The crystal structure of $[CuCl_2(C_{12}H_{12}N_2)]$ is reported. The complex is monomeric, with the Cu atom coordinated to two N atoms and two Cl^- ions. The compound displays simple paramagnetic behaviour.

Comment

Copper(II) shows a great 'plasticity' in its coordination geometries, as expected for an ion with Jahn–Teller active ground states (Bersuker, 1984). This fact has permitted the magneto-structural studies of Cu^{II} complexes with heterocyclic amines. Continuing the characterization of complexes with di(2-pyridyl)methane ligands, we have prepared $[Cu(MeDPM)_2Cl_2]$, (I), where MeDPM is 6-methyl-2-(2-pyridylmethyl)pyridine. While the chloro and bromo di(2-pyridyl)copper(II) complexes are dimeric in nature and show a ferromagnetically coupled ground state (Spodine, Manzur, Peña & Garland, 1986; Spodine *et al.*, 1993), the title complex is found to be monomeric, with simple paramagnetic behaviour.



(I)

The Cu atom is coordinated to two N atoms from the bidentate MeDPM ligand and to two Cl^- ions [Cu—N 2.010 (4) and 2.013 (4) Å, Cu—Cl 2.224 (2) and 2.242 (2) Å]. The dihedral angle between the N—Cu—N and Cl—Cu—Cl planes is 48°, half-way between that characterizing a square-planar arrangement (0°) and that of a regular tetrahedron (90°). Inspection of Fig. 1 suggests that the driving force for this distortion is the steric repulsion between the methyl group attached to C11 and the chloro ligand. This argument is supported by comparison with the closely related structure of dichlorodi-μ-chloro-bis(dipyridylmethane)dicopper(II) (Spodine *et al.*, 1993), which differs from (I) only in the absence of the methyl substituent; in this complex the homologous dihedral angle spans 29° and is much closer to that of a planar arrangement. This fact in turn seems to enable the inclusion of a fifth ligand in the Cu coordination sphere (a symmetry-related Cl atom), leading to the formation of a dimer. The monomeric nature of (I) then seems to be a direct consequence of the presence of the methyl group in the organic chelate. Another interesting difference between these two structures is the dihedral angle between the two pyridyl rings of the organic ligand, which in the dimeric complex is 64.8° (one of the largest found), while in (I) it has a value of 56.2 (2)°, the smallest reported (Garland, Grandjean, Spodine & Manzur, 1987; Garland, Le Marouille, Spodine & Manzur, 1986; Spodine *et al.*, 1993; Spodine *et al.*, 1991).

There are three short non-bonding H···Cl contacts below 3.0 Å in the structure: C4—H4*A*···Cl2$^{\mathrm{i}}$ *ca* 2.95 Å, C6—H6*B*···Cl1$^{\mathrm{i}}$ *ca* 2.91 Å and C8—H8*A*···Cl1$^{\mathrm{i}}$ *ca* 2.97 Å [symmetry code: (i) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$] (Fig. 2).

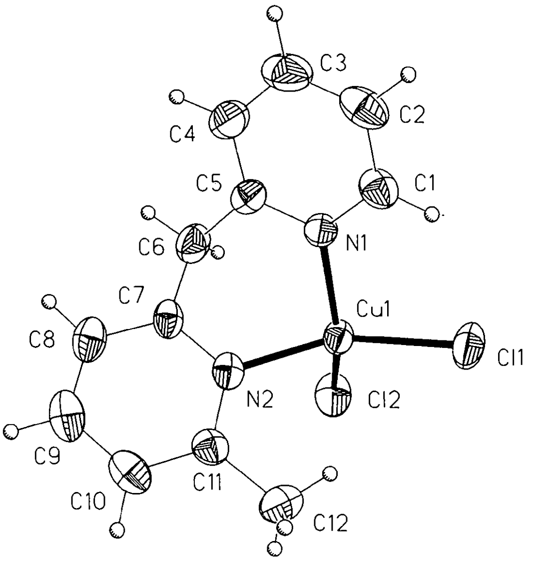


Fig. 1. View of the molecule showing the labelling of the non-H atoms. Displacement ellipsoids are plotted at the 50% probability level.

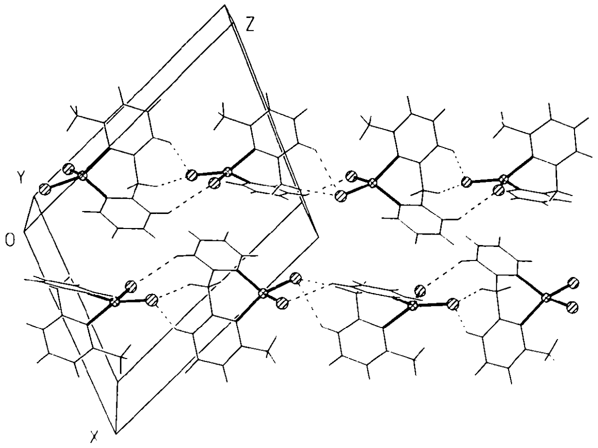


Fig. 2. Packing of the molecules showing the short non-bonding intermolecular contacts.

Experimental

Crystal data

$[\mathrm{CuCl_2(C_{12}H_{12}N_2)}]$
$M_r = 318.68$
Monoclinic
$P2_1/n$
$a = 12.125\ (2)$ Å
$b = 7.593\ (2)$ Å
$c = 15.216\ (3)$ Å
$\beta = 112.86\ (3)°$
$V = 1290.8\ (5)$ Å^3
$Z = 4$
$D_x = 1.640$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.5–12.5°$
$\mu = 2.083$ mm^{-1}
$T = 293\ (2)$ K
Plate
0.42 × 0.32 × 0.06 mm
Green

Data collection

Siemens $R3m$ diffractometer
$\theta/2\theta$ scans (4.19–29.3° min^{-1})
Absorption correction: none
1793 measured reflections
1702 independent reflections
1387 observed reflections [$I > 2\sigma(I)$]

$R_{\mathrm{int}} = 0.0263$
$\theta_{\mathrm{max}} = 22.76°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 8$
$l = -16 \rightarrow 15$
2 standard reflections monitored every 98 reflections intensity variation: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0476$
$wR(F^2) = 0.1137$
$S = 1.163$
1702 reflections
154 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0812P)^2 + 0.1467P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\mathrm{max}} = 0.094$
$\Delta\rho_{\mathrm{max}} = 0.715$ e Å^{-3}
$\Delta\rho_{\mathrm{min}} = -0.846$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cu1	0.02025 (5)	0.13736 (8)	0.27088 (4)	0.0348 (3)
Cl1	−0.06963 (12)	−0.0144 (2)	0.13690 (9)	0.0525 (4)
Cl2	−0.02378 (13)	0.4119 (2)	0.21378 (9)	0.0506 (4)
N1	0.1573 (3)	−0.0303 (6)	0.3314 (3)	0.0350 (10)
N2	0.0250 (3)	0.1991 (5)	0.4009 (3)	0.0332 (10)
C1	0.1621 (5)	−0.1945 (7)	0.2977 (4)	0.0412 (13)
C2	0.2583 (5)	−0.3027 (8)	0.3413 (4)	0.051 (2)
C3	0.3487 (5)	−0.2486 (9)	0.4220 (4)	0.056 (2)
C4	0.3433 (5)	−0.0819 (9)	0.4591 (4)	0.050 (2)
C5	0.2471 (4)	0.0254 (7)	0.4113 (3)	0.0376 (13)
C6	0.2378 (5)	0.2084 (7)	0.4463 (4)	0.0445 (13)
C7	0.1316 (4)	0.2298 (7)	0.4722 (3)	0.0351 (12)
C8	0.1399 (5)	0.2790 (7)	0.5618 (4)	0.0448 (14)
C9	0.0397 (6)	0.2996 (8)	0.5802 (4)	0.0513 (15)
C10	−0.0690 (5)	0.2687 (7)	0.5076 (4)	0.0465 (14)
C11	−0.0751 (4)	0.2173 (7)	0.4181 (3)	0.0378 (12)
C12	−0.1897 (5)	0.1826 (9)	0.3371 (4)	0.053 (2)

Table 2. *Selected geometric parameters* (Å, °)

Cu1—N1	2.010 (4)	C3—C4	1.397 (9)
Cu1—N2	2.013 (4)	C4—C5	1.376 (7)
Cu1—Cl1	2.224 (2)	C5—C6	1.508 (8)
Cu1—Cl2	2.242 (2)	C6—C7	1.496 (7)
N1—C5	1.346 (6)	C7—C8	1.379 (7)
N1—C1	1.359 (7)	C8—C9	1.358 (8)
N2—C11	1.345 (6)	C9—C10	1.371 (8)
N2—C7	1.346 (6)	C10—C11	1.391 (7)
C1—C2	1.370 (8)	C11—C12	1.479 (7)
C2—C3	1.353 (8)		
N1—Cu1—N2	89.7 (2)	C5—C4—C3	119.0 (5)
N1—Cu1—Cl1	96.64 (12)	N1—C5—C4	120.9 (5)
N2—Cu1—Cl1	146.99 (13)	N1—C5—C6	117.6 (4)
N1—Cu1—Cl2	142.80 (12)	C4—C5—C6	121.5 (5)
N2—Cu1—Cl2	94.36 (12)	C7—C6—C5	112.5 (4)
Cl1—Cu1—Cl2	99.70 (6)	N2—C7—C8	121.5 (5)
C5—N1—C1	119.4 (4)	N2—C7—C6	115.1 (4)
C5—N1—Cu1	116.7 (3)	C8—C7—C6	123.4 (4)
C1—N1—Cu1	123.9 (3)	C9—C8—C7	120.5 (5)
C11—N2—C7	118.8 (4)	C8—C9—C10	118.1 (5)
C11—N2—Cu1	122.2 (3)	C9—C10—C11	120.4 (5)
C7—N2—Cu1	119.0 (3)	N2—C11—C10	120.7 (5)
N1—C1—C2	121.4 (5)	N2—C11—C12	116.5 (4)
C3—C2—C1	119.6 (6)	C10—C11—C12	122.8 (5)
C2—C3—C4	119.6 (5)		

Data collection and cell refinement: *P3/P4-PC Diffractometer Program* (Siemens, 1991). Data reduction: *SHELXTL/PC XDISK* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXTL/PC XS*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1994). Molecular graphics: *SHELXTL/PC XP*. Software used to prepare material for publication: *SHELXL93 CIFTAB*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: BK1029). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Diamminesilver(I) Bis(biuretato-N^1,N^5)-nickelate(II) Hexahydrate

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Abstract

The title compound, $[Ag(NH_3)_2]_2[Ni(C_2H_3N_3O_2)_2].6H_2O$, consists of planar $[Ni(biu)_2]^{2-}$ anions (biu is the biuret ligand), with square-planar coordination of the Ni atom, and almost linear diamminesilver(I) cations. The cations and anions are stacked in a columnar fashion along the *a* axis. The packing is determined by intermolecular hydrogen bonds involving the cations, anions and water molecules.

Comment

Biuret easily forms complexes with metal ions and the structures of the copper(II) and cobalt(III) complexes (Birker & Beurskens, 1974; Freeman, Smith & Taylor, 1961; Pajunen & Pajunen, 1982) have been determined. However, only a few nickel(II)–biuretato complexes have been discussed (Traube, 1922) and no crystal

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