Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.158$
$S = 0.931$
8182 reflections
489 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.353$ e Å^{-3}
$\Delta\rho_{\min} = -0.355$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Mg1—O4	2.048 (3)	Mg—O4*A*	2.064 (3)
Mg1—O3	2.050 (3)	C14*A*—O1*A*	1.257 (4)
Mg1—O5	2.076 (3)	C14*A*—O2*A*	1.262 (4)
Mg—O3*A*	2.048 (3)	C14—O1	1.255 (4)
Mg—O5*A*	2.056 (3)	C14—O2	1.260 (4)
O4—Mg1—O3	87.23 (12)	O5*A*—Mg—O4*A*	89.34 (12)
O4—Mg1—O5	89.59 (13)	C7*A*—N1*A*—C1*A*	123.3 (3)
O3—Mg1—O5	90.91 (13)	O1*A*—C14*A*—O2*A*	123.5 (4)
O3*A*—Mg—O5*A*	90.14 (12)	C7—N1—C1	122.4 (3)
O3*A*—Mg—O4*A*	88.91 (12)	O1—C14—O2	124.3 (4)
C1*A*—N1*A*—C7*A*—C12*A*	−59.1 (6)		
C1—N1—C7—C12	65.2 (5)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl2	2.54 (4)	2.990 (4)	119 (4)
N1—H1···O1	2.24 (4)	2.898 (4)	143 (5)
N1*A*—H1*A*···Cl2*A*	2.40 (4)	2.969 (4)	130 (4)
N1*A*—H1*A*···O1*A*	2.36 (4)	2.940 (4)	131 (4)
O3—H11···O1*A*	2.11 (5)	2.902 (4)	160 (5)
O3*A*—H11*A*···O1^{i}	1.99 (5)	2.831 (4)	157 (5)
O3—H12···O2^{ii}	1.87 (4)	2.730 (4)	173 (5)
O3*A*—H12*A*···O9*A*^{iii}	1.95 (5)	2.769 (4)	159 (5)
O4—H13···O9*A*^{iv}	1.89 (5)	2.713 (4)	167 (5)
O4*A*—H13*A*···O2^{v}	1.92 (4)	2.852 (4)	166 (4)
O4—H14···O9	1.99 (4)	2.879 (4)	159 (4)
O4*A*—H14*A*···O2*A*	1.96 (4)	2.793 (4)	171 (5)
O5—H15···O2*A*^{vi}	1.87 (5)	2.758 (4)	167 (4)
O5*A*—H15*A*···O9	1.94 (5)	2.734 (4)	169 (5)
O9—H23···O1	1.78 (4)	2.834 (4)	172 (4)
O9*A*—H23*A*···O1*A*	1.64 (4)	2.673 (4)	170 (4)
O9—H24···O2^{vii}	1.90 (4)	2.776 (4)	170 (4)
O9*A*—H24*A*···O2*A*^{iv}	2.08 (4)	2.804 (4)	163 (5)

Symmetry codes: (i) $x, 1+y, z$; (ii) $1+x, y, z$; (iii) $x-1, y, z$; (iv) $1-x, 1-y, -z$; (v) $-x, 1-y, -z$; (vi) $x, y-1, z$; (vii) $-x, -y, -z$.

The N—H and water H atoms were located from a difference synthesis and were refined isotropically. The remaining H atoms were placed in calculated positions and refined riding on their parent atoms.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *ORTEP*II (Johnson, 1976).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1405). Services for accessing these data are described at the back of the journal.

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trans-Bis(acetato-*O*)bis(2-amino-6-methylpyridine-*N*)copper(II)

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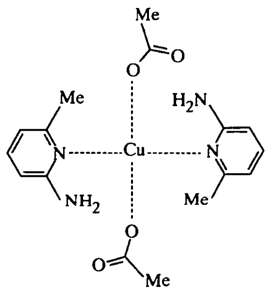
Abstract

In the title compound, $[Cu(C_2H_3O_2)_2(C_6H_8N_2)_2]$, the Cu atom is surrounded by two O atoms from the two monodentate acetate ligands and by two pyridine N atoms from the two 2-amino-6-methylpyridine ligands in a *trans* square-planar arrangement. The Cu—O and Cu—N distances are 1.952 (2) and 2.046 (2) Å, respectively. The complex has an inversion centre. The remaining carboxylate O atoms are located at 2.764 (3) Å from the Cu atom, above and below this plane. The coordination around the Cu atom can therefore be described as (4+2). The pyridine ring is twisted 73.2 (1)° with respect to the plane formed by the Cu, the N and the more strongly bonded O atoms.

Comment

As part of a continuing study of copper(II) carboxylates, we report here the structure of *trans*-bis(acetato-*O*)bis(2-amino-6-methylpyridine-*N*)copper(II), $[Cu(C_2H_3O_2)_2(C_6H_8N_2)_2]$, (I), which is the first copper complex with 2-amino-6-methylpyridine as a ligand [survey of the Cambridge Structural Database (Allen & Kennard, 1993)]. Recently, the crystal structure, spectroscopic and magnetic studies of *cis*-bis(acetato-*O*)bis(2-aminopyridine-*N*)copper(II) were reported (Grobelny *et al.*, 1995), in which the 2-aminopyridine ligands are arranged *cis* to each other. However, in monomeric complexes with a methyl group instead of an amino group in the *ortho* position, the *trans* arrangement was observed (Borel *et al.*, 1981; Heimer & Ahmed, 1982; Busnot *et al.*, 1983). The crystal structure of complex (I) was determined in order to find out how the ligands are arranged around the Cu atom and to compare the results with related structures.



(I)

The stereochemistry of the title molecule is essentially four-coordinate with a CuN_2O_2 chromophore. Similar coordination was observed in some other monomeric copper acetates containing substituted pyridines with methyl groups on the 2,6-positions (Borel *et al.*, 1981; Heimer & Ahmed, 1982; Busnot *et al.*, 1983). In the present complex, the 2-amino-6-methylpyridine ligand is orientated in such a way that the amino group blocks further bonding from the Cu atom to the second carboxylate oxygen (O2), but allows hydrogen bonding between the amino group and O2 [2.952 (3) Å]. The Cu—O2 distance of 2.764 (3) Å is comparable with the value found in *trans*-diacetatobis(collidine)copper(II) [2.772 (Heimer & Ahmed, 1982) and 2.788 (3) Å (Busnot *et al.*, 1983)]; it is substantially longer than the Cu—O1 distance of 1.952 (2) Å but shorter than the sum of the van der Waals radii (2.80 Å), which indicates a weak interaction between the two atoms. Therefore, the (4+2) coordination is proposed. The shorter C1—O2 bond length of 1.235 (3) Å suggests more double-bond character for C1—O2 compared with the longer C1—O1 distance of 1.272 (3) Å.

The twist of the pyridine ring is 73.2 (1)° with respect to the Cu/O1/N1 plane, thus avoiding steric interactions with the semi-coordinated O atom of the acetate group. There is also an intermolecular hydrogen bond between the amino group and the semi-coordinated O atom at ($-x+1$, $-y$, $-z+1$), which links the monomeric Cu^{II} complex molecules [2.922 (4) Å; see Table 2]. A similar intramolecular hydrogen bond is present in *cis*-bis(acetato-*O*)bis(2-aminopyridine-*N*)copper(II), but neighbouring molecules are linked through the amino group and the more strongly bonded acetate O atom. These different hydrogen-bonding schemes could be the reason for the different arrangement of ligands around the Cu atom in these two complexes.

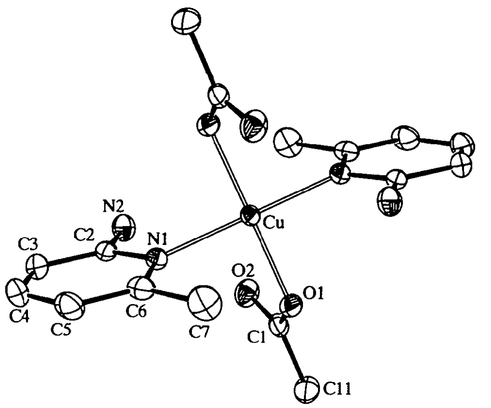


Fig. 1. An *ORTEP*II (Johnson, 1971) view of the title complex with the atomic numbering scheme. Anistropic displacement ellipsoids are drawn at the 30% probability level and H atoms have been omitted for clarity.

Experimental

$Cu_2(C_2H_3O_2)_4\cdot 2H_2O$ (0.4 g, 1 mmol) was dissolved in acetonitrile (40 ml) by heating and stirring. 2-Amino-6-methylpyridine (0.43 g, 4 mmol) was then added. Dark-violet crystals of (I) appeared after a few days in the refrigerator.

Crystal data

$[Cu(C_2H_3O_2)_2(C_6H_8N_2)_2]$
M_r = 397.92
Monoclinic
$P2_1/n$
a = 7.887 (9) Å
b = 8.358 (4) Å
c = 13.98 (1) Å
β = 103.49 (6)°
V = 896.1 (13) $Å^3$
Z = 2
D_x = 1.475 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 50 reflections
θ = 7.83–12.67°
μ = 1.247 mm^{-1}
T = 293 (2) K
Prismatic
0.22 × 0.21 × 0.14 mm
Dark violet

Data collection

Enraf–Nonius CAD-4 diffractometer
Variable $\omega/2\theta$ scan
Absorption correction: by integration (*NRCVAX*; Gabe *et al.*, 1989)
T_{min} = 0.775, T_{max} = 0.832
8329 measured reflections
2163 independent reflections
1428 reflections with $I > 2\sigma(I)$

R_{int} = 0.058
θ_{max} = 27.97°
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -18 \rightarrow 18$
3 standard reflections every 600 reflections
intensity decay: 11%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.086$
$S = 1.035$
2163 reflections
118 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2 + 0.2858P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.455$ e Å^{-3}
$\Delta\rho_{\min} = -0.503$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0041 (15)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Cu—O1	1.952 (2)	C1—C11	1.505 (4)
Cu—O2	2.764 (3)	C2—C3	1.404 (4)
Cu—N1	2.046 (2)	C3—C4	1.357 (5)
O1—C1	1.272 (3)	C5—C4	1.377 (5)
N1—C2	1.350 (3)	C6—C5	1.377 (4)
N1—C6	1.361 (3)	C6—C7	1.487 (4)
O2—C1	1.235 (3)	N2—C2	1.356 (3)
O1—Cu—N1	91.59 (9)	O1—C1—C11	116.1 (3)
O1—Cu—O2	52.38 (7)	N1—C2—N2	117.7 (2)
N1—Cu—O2	86.98 (9)	N1—C2—C3	121.3 (2)
C1—O1—Cu	111.04 (16)	N2—C2—C3	121.1 (2)
C2—N1—C6	119.1 (2)	N1—C6—C5	120.9 (3)
C2—N1—Cu	119.04 (16)	N1—C6—C7	117.4 (2)
C6—N1—Cu	121.10 (18)	C5—C6—C7	121.7 (3)
C1—O2—Cu	73.39 (16)	C4—C3—C2	118.8 (3)
O2—C1—O1	123.2 (2)	C4—C5—C6	119.7 (3)
O2—C1—C11	120.7 (3)	C3—C4—C5	120.1 (3)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···O2	0.86	2.36	2.952 (3)	126
N2—H2···O2^{i}	0.86	2.14	2.922 (4)	151

Symmetry code: (i) $1 - x, -y, 1 - z$.

H atoms were included at calculated positions using *SHELXL*97 (Sheldrick, 1997) and refined using a riding model, with isotropic displacement parameters taken from the parent atoms and multiplied by 1.5 for methylenic and methyl H atoms, and by 1.2 for amino and phenyl H atoms.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1994). Cell refinement: *XRAY*76 (Stewart *et al.*, 1976). Data reduction: *NRCVAX* (Gabe *et al.*, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*97. Molecular graphics: *ORTEP*II (Johnson, 1971). Software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1255). Services for accessing these data are described at the back of the journal.

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Di-μ-chloro-bis(aquachlorodimethyltin)–dichlorodimethyl[2,4,6-tris(2-pyridyl)-1,3,5-triazine]tin (1/2)

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Abstract

In the centrosymmetric title compound, $[Sn_2(CH_3)_4Cl_4(H_2O)_2]\cdot 2[Sn(CH_3)_2Cl_2(C_{18}H_{12}N_6)]$, the Sn atom of the dichlorodimethyl[2,4,6-tris(2-pyridyl)-1,3,5-triazine]tin moiety is seven-coordinate in a *trans*-$C_2SnCl_2N_3$ pentagonal bipyramidal geometry [C—Sn—C 157.0 (2)°] and that of the water-coordinated di-μ-chloro-bis(aquachlorodimethyltin) moiety is six-coordinate in a *trans*-C_2SnCl_3O octahedral geometry [C—Sn—C 146.2 (2)°]. In the water-coordinated moiety, the Sn···Cl bridge [3.425 (2) Å] is *trans* to the Sn—O$_{\text{water}}$ bond [2.391 (4) Å]; each water molecule is linked to a free pyridyl N atom of the heterocycle by only one hydrogen bond [O···N 2.821 (6) Å].

Comment

2,2′:6′,2″-Terpyridyl when treated with dimethyltin diisothiocyanate affords a seven-coordinate *N*,*N*′,*N*″-

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