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(−)-Sparteine copper(II) diacetate[1]

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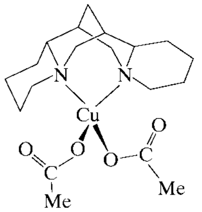
The chiral nitrogen-chelating alkaloid (−)-sparteine acts as a bidentate ligand, reacting with copper(II) acetate in ethanol to form the title complex, $[Cu(CH_3COO)_2(C_{15}H_{26}N_2)]$, with the two acetate groups occupying the remaining coordination sites in a monodentate fashion to produce a distorted four-coordinate tetrahedral structure. The dihedral angle between the N—Cu—N and O—Cu—O planes is 45.8 (3)°.

Comment

Previously, we determined the crystal structures of (−)-sparteine copper(II) dinitrate and (−)-sparteine copper(II) dinitrite (Choi *et al.*, 1995; Lee, Choi *et al.*, 1998). The molecules of (−)-sparteine copper(II) dinitrate are mixed, with both four- and five-coordinate species in one crystalline phase and only the four-coordinate species in the other (Choi *et al.*, 1995). However, the copper(II) ion in (−)-sparteine copper(II) dinitrite is exclusively five-coordinate, with a highly distorted square-pyramidal geometry (Lee, Choi *et al.*, 1998). The acetate ion, like the nitrate or nitrite ions, can coordinate to a metal in either a mono- or a bidentate fashion, and we expected that (−)-sparteine copper(II) diacetate, (I), might show either a four- or five-coordinate geometry around the copper(II) ion. The copper(II) ion in this complex is found to be exclusively four-coordinate, with a distorted tetrahedral geometry.



(I)

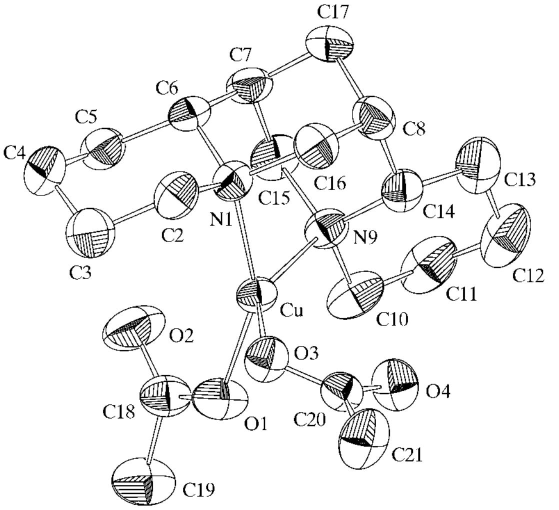


Figure 1
ZORTEP (Zsolnai, 1996) diagram of (I) showing the atom-numbering scheme and 40% probability ellipsoids. H atoms have been omitted for clarity.

The N1—Cu—N9 plane in (I) is twisted by 45.8 (3)° from the O1—Cu—O3 plane. The respective bond lengths of Cu—O1 and Cu—O3 are 1.927 (5) and 1.973 (4) Å, and these values are similar to those found in strongly coordinated anions (Togni *et al.*, 1990; Choi *et al.*, 1995; Lopez *et al.*, 1998; Lee, Choi *et al.*, 1998; Lee, Oh *et al.*, 1998). The Cu···O2 and Cu···O4 distances, which are 3.061 (5) and 2.701 (5) Å, respectively, are too well separated to be considered as bonding interactions. The Cu—N bond distances of 2.010 (6) and 2.049 (6) Å are comparable to those found in other (−)-sparteine copper(II) complexes (Togni *et al.*, 1990; Choi *et al.*, 1995; Lopez *et al.*, 1998; Lee, Choi *et al.*, 1998).

The O—C—O bond angles in (I) are observed to be 124.9 (8) and 124.0 (5)°, and these are similar to the value in the free acetate (Hsu & Nordman, 1983) and are much greater than the O—N—O bond angles of coordinated nitrates or nitrites (Choi *et al.*, 1995; Lee, Choi *et al.*, 1998). Furthermore, if the acetate ion chelates to the copper(II) centre, double-bond character will be developed at the *endo*-position of the chelate ring and this will require the O—C—O bond angle to be more open. The chelated acetate will cause greater ring strain than the chelated nitrite or nitrate does. As a result, the acetate seems to prefer to coordinate to the copper(II) ion in a monodentate fashion.

Experimental

Complex (I) was prepared by the direct reaction of copper(II) acetate with a stoichiometric amount of (−)-sparteine in ethanol–triethylorthoformate (5:1 *v*/*v*) solution. Single crystals were obtained by recrystallization at about 278 K from a dichloromethane–triethylorthoformate (4:1 *v*/*v*) solution under carbon tetrachloride vapor.

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[1] Alternative name: (1,3,4,7,7a,8,9,10,11,13,14,14a-dodecahydro-7,14-methano-2*H*,6*H*-dipyrido[1,2-*a*:1′,2′-*e*][1,5]diazocine-*N*,*N*′)bis(acetato-*O*,*O*′)-copper(II).

Crystal data

$[Cu(C_2H_3O_2)_2(C_{15}H_{26}N_2)]$
$M_r = 416.01$
Monoclinic, $P2_1$
$a = 8.0507$ (8) Å
$b = 12.0791$ (12) Å
$c = 10.2946$ (8) Å
$\beta = 97.954$ (8)°
$V = 991.47$ (16) Å^3
$Z = 2$

$D_x = 1.393$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 7.32$–$13.04°$
$\mu = 1.127$ mm^{-1}
$T = 293$ (2) K
Cube, blue
0.60 × 0.45 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.543$, $T_{\max} = 0.754$
2013 measured reflections
1838 independent reflections
1626 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.020$
$\theta_{\max} = 24.97°$
$h = 0 \rightarrow 9$
$k = -2 \rightarrow 14$
$l = -12 \rightarrow 12$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.101$
$S = 1.315$
1838 reflections
230 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0136P)^2 + 1.9871P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.51$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu—O1	1.927 (5)	C18—O2	1.229 (9)
Cu—O3	1.973 (4)	C18—O1	1.266 (10)
Cu—N1	2.010 (6)	C20—O4	1.216 (8)
Cu—N9	2.049 (6)	C20—O3	1.278 (7)
O1—Cu—O3	92.4 (3)	O3—Cu—N9	142.9 (3)
O1—Cu—N1	151.8 (2)	N1—Cu—N9	90.1 (2)
O3—Cu—N1	91.7 (2)	O2—C18—O1	124.9 (8)
O1—Cu—N9	103.1 (3)	O4—C20—O3	124.0 (5)

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989); data reduction: *Xtal*3.2 *Reference Manual* (Hall *et al.*, 1992); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1996); 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: TA1266). Services for accessing these data are described at the back of the journal.

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