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Redetermination of tetrakis(μ-acetato-κ^2O:O')-bis[(2-aminopyridine-κN)copper(**II**)]

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Key indicators

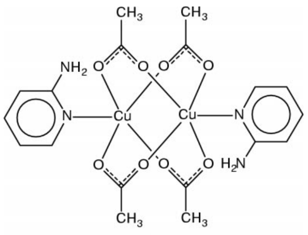
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.025
wR factor = 0.067
Data-to-parameter ratio = 15.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The crystal structure of the title compound, $[Cu_2(C_2H_3O_2)_4(C_5H_6N_2)_2]$, previously reported by Antsyshkina, Porai-Koshits, Ostrikova, Garnovskii, Sadimenko & Osipov [*Koord. Khim.* (1987), **13**, 836–842] has been redetermined from new intensity data, with appreciably improved precision and successful location of all H atoms. The dinuclear copper unit has crystallographic inversion symmetry with a Cu$\cdots$Cu distance of 2.6771 (4) Å. The crystal packing is characterized by C—H$\cdots$O hydrogen bonds.

Comment

The crystal structure of the title compound, (I), comprises dinuclear $[Cu_2(C_2H_3O_2)_4(C_5H_6N_2)_2]$ units. The molecule is located on a crystallographic inversion centre. The Cu$\cdots$Cu distances observed in dimeric copper(II) acetate complexes varies from 2.575 to 2.709 Å (*ConQuest*; CCDC, 2003). Thus, the Cu$\cdots$Cu($-x$, $-y$, $-z$) distance of 2.6771 (4) Å found in the present compound is towards the higher limit of this range. The deviation of the Cu atom from the mean plane formed by the four equatorial O atoms towards the apical 2-aminopyridine ligand is 0.2228 (7) Å.



(I)

The observed Cu—O1 bond length of 1.9883 (13) Å is longer than the other Cu—O distances of 1.9773 (13), 1.9803 (13) and 1.9811 (13) Å. The elongation of this Cu—O distance may be due to the N1—H1$\cdots$O1 intramolecular hydrogen bond. The remaining acetate O atoms are involved in weak C—H$\cdots$O intra- (O2) and intermolecular (O3 and O4) interactions. The hydrogen-bonding geometry is listed in Table 2. The atom H2 of the NH_2 group does not contribute to the hydrogen-bonding network.

The planes of the two independent acetate groups are perpendicular within experimental error. The pyridine ring is twisted by 19.6 (1)° with respect to the the acetate (C1/C2/O1/O2) ligand mean plane.

The geometric parameters of the present structure agree well with those previously reported by Antsyshkina *et al.* (1987), but with improved precision. In addition, the positions of the all H atoms have been determined.

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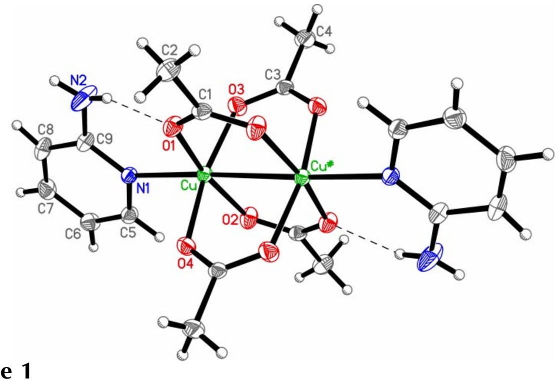

Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate the hydrogen bonds [symmetry code: (#) $-x$, $-y$, $-z$].

Experimental

The title complex was prepared by dissolving cupric acetate [1 mmol, $Cu(CH_3COO)_2 \cdot H_2O$] in 30 ml of water with a few drops of acetic acid to clear the solution. After heating to boiling, 2-aminopyridine (1 mmol, $C_5H_6N_2$) in 20 ml of water was added. Green crystals of (I) were grown by slow evaporation over several days.

Crystal data

$[Cu_2(C_2H_3O_2)_4(C_5H_6N_2)_2]$
$M_r = 551.51$
Monoclinic, $P2_1/c$
$a = 7.4498$ (3) Å
$b = 19.6500$ (7) Å
$c = 8.1251$ (4) Å
$\beta = 114.316$ (4)°
$V = 1083.91$ (9) Å^3
$Z = 2$

$D_x = 1.690$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8156 reflections
$\theta = 2.8$–27.5°
$\mu = 2.02$ mm^{-1}
$T = 293$ (2) K
Prism, green
$0.36 \times 0.30 \times 0.12$ mm

Data collection

Kuma KM4CCD diffractometer
ω scans
Absorption correction: numerical (*XRED*; Stoe & Cie, 1999)
$T_{min} = 0.500$, $T_{max} = 0.782$
12569 measured reflections
2483 independent reflections

2143 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$
$\theta_{max} = 27.5°$
$h = -9 \rightarrow 8$
$k = -25 \rightarrow 25$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.067$
$S = 1.02$
2483 reflections
165 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0394P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu—O1	1.9883 (13)	Cu—O4	1.9773 (13)
Cu—O2	1.9803 (13)	Cu—N1	2.2239 (15)
Cu—O3	1.9811 (13)		
O1—Cu—O2	167.03 (6)	O3—Cu—O4	167.15 (5)
O1—Cu—O3	89.18 (6)	N1—Cu—O1	97.97 (6)
O1—Cu—O4	89.32 (6)	N1—Cu—O2	95.00 (5)
O2—Cu—O3	89.14 (6)	N1—Cu—O3	97.05 (5)
O2—Cu—O4	89.46 (6)	N1—Cu—O4	95.80 (5)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H1$\cdots$O1	0.94 (3)	2.00 (3)	2.878 (3)	155 (3)
C5—H5$\cdots$O2	0.93	2.49	3.103 (3)	124
C8—H8$\cdots$O3^{i}	0.93	2.56	3.471 (3)	167
C4—H43$\cdots$O4^{ii}	0.96	2.53	3.454 (3)	162

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

All H atoms were initially located in a difference Fourier synthesis. The aromatic H atoms were positioned with idealized geometry, with C—H = 0.93 Å. The positions of the methyl H atoms were idealized with C—H = 0.96 Å and refined using a riding model (including free rotation about C—C bonds). The amine H atoms and isotropic displacement parameters of all H atoms were refined freely.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2004); program(s) used to solve structure: *SIR*2002 (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2003); molecular graphics: *SHELXTL*; software used to prepare material for publication: *PLATON* (Spek, 2003).

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