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Tetra-μ-acetato-bis[aquacopper(II)]–4,5-diazafluoren-9-one (1/2)

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In the title compound, $[Cu_2(CH_3CO_2)_4(H_2O)_2]\cdot 2C_{11}H_6N_2O$, the dinuclear copper complex lies on a center of symmetry located midway between the two Cu atoms. The two Cu atoms are bridged by four acetate groups. The coordinated water molecules interact with the $C_{11}H_6N_2O$ molecules and bridging acetate ligands by hydrogen bonds, giving rise to a linear chain structure.

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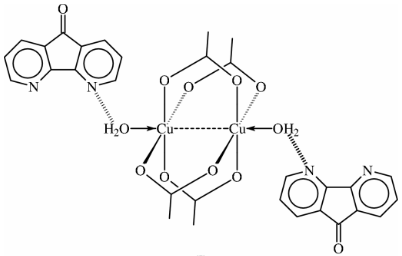
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.050
wR factor = 0.114
Data-to-parameter ratio = 16.5

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

Comment

4,5-Diazafluoren-9-one is not as good a chelating reagent as, for example, the diimine 2,2′-bipyridine (Wu *et al.*, 2002). In the context of its ability to bind to Cu, the reagent, whose crystal structure has been determined twice previously (Fun *et al.*, 1995; Ravikumar & Lakshmi, 1994), chelates to copper halides (Balagopalakrishna *et al.*, 1992; Menon & Rajasekharan, 1998). With copper perchlorate, the ligand binds directly to the Cu center to form a bis-chelate compound (Gu *et al.*, 2002; Menon & Rajasekharan, 1998; Zhao *et al.*, 2003; Zhang *et al.*, 2003), as well as a complex having both chelating and free ligands (Yang *et al.*, 2001).



(I)

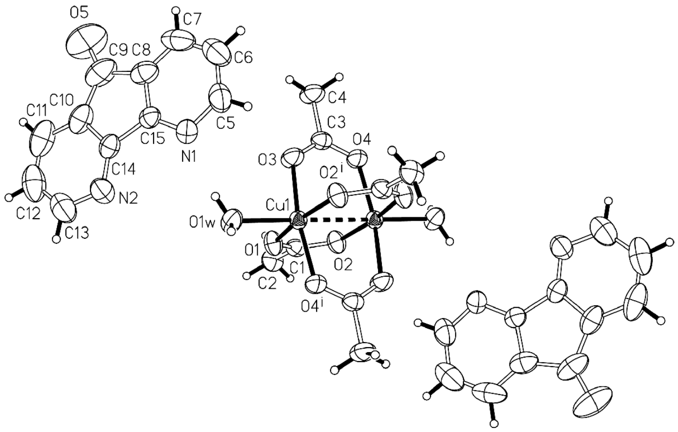


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I); displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. [Symmetry code: (i) $1-x, 1-y, 1-z$.]

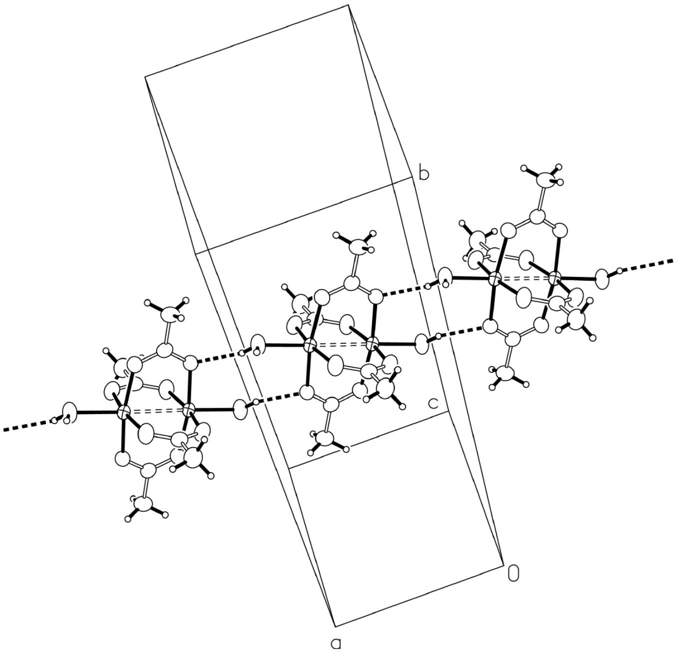


Figure 2
*ORTEP*II (Johnson, 1976) plot of the hydrogen-bonded (dashed lines) diaquatetra-μ-acetato-dicopper chain.

The ligand binds only indirectly, through the coordinated water molecules, to the tetraacetate complex, (I) (Fig. 1). The centrosymmetric dinuclear complex retains the conformation it possesses in tetra-μ-acetato-dicopper itself (Ferguson & Glidewell, 2003). This propagates as a linear chain whose repeat units are held together by a hydrogen bond involving the water molecule (Fig. 2). The water molecule uses its other H atom to interact with one N atom of the heterocycle (Table 2). The packing is probably not compact, as noted from the sum of the atomic volumes of the dinuclear complex in tetra-μ-acetato-dicopper (417.8 Å^3) and that of the free ligand (348.1 Å^3).

Experimental

A drop of tris(2-aminoethyl)amine (~ 0.05 g) was added to a methanol solution of copper acetate dihydrate (0.20 g, 1 mmol). Bis(4,5-diazafluoren-9-one) (0.09 g, 0.5 mmol) dissolved in methanol was added and the solution was refluxed for 1 h. The solution was left aside for several days for the crystals (m.p. 478–479 K) to separate out. CHN analysis calculated for $C_{30}H_{28}Cu_2N_4O_{12}$: C 47.14, N 7.33, H 3.67%; found C 47.59, N 7.40, H 3.57%.

Crystal data

$[Cu_2(C_2H_3O_2)_4(H_2O)_2]\cdot 2C_{11}H_6N_2O$
$M_r = 763.64$
Monoclinic, $P2_1/a$
$a = 7.4661\ (3)$ Å
$b = 25.951\ (1)$ Å
$c = 8.2342\ (3)$ Å
$\beta = 92.604\ (1)^\circ$
$V = 1593.7\ (1)$ Å^3
$Z = 2$
$D_x = 1.591$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8182 reflections
$\theta = 2.5–27.7^\circ$
$\mu = 1.40$ mm^{-1}
$T = 298\ (2)$ K
Wedge, blue
0.32 × 0.24 × 0.21 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.574$, $T_{\max} = 0.746$
17 996 measured reflections
3753 independent reflections
3120 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.044$
$\theta_{\max} = 28.2^\circ$
$h = -9 \rightarrow 9$
$k = -33 \rightarrow 33$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.114$
$S = 1.12$
3753 reflections
227 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.39$ e Å^{-3}
$\Delta\rho_{\min} = -0.60$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.963 (2)	Cu1—O4$^{\rm i}$	1.967 (2)
Cu1—O2$^{\rm i}$	1.976 (2)	Cu1—O1*w*	2.137 (2)
Cu1—O3	1.969 (2)		
O1—Cu1—O2$^{\rm i}$	168.6 (1)	O2$^{\rm i}$—Cu1—O4$^{\rm i}$	89.1 (1)
O1—Cu1—O3	88.9 (1)	O2$^{\rm i}$—Cu1—O1*w*	97.3 (1)
O1—Cu1—O4$^{\rm i}$	89.4 (1)	O3—Cu1—O4$^{\rm i}$	168.7 (1)
O1—Cu1—O1*w*	94.1 (1)	O3—Cu1—O1*w*	96.4 (1)
O2$^{\rm i}$—Cu1—O3	90.3 (1)	O4$^{\rm i}$—Cu1—O1*w*	94.9 (1)

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*w*—H1*w*1···N1	0.84 (1)	2.01 (1)	2.834 (3)	170 (4)
O1*w*—H1*w*2···O2$^{\rm ii}$	0.84 (1)	2.06 (2)	2.843 (3)	156 (3)

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

The C-bound H atoms were positioned geometrically [C—H = 0.98 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl H atoms, and C—H = 0.93 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for aromatic H atoms] and were allowed to ride on their parent atoms in the riding-model approximation. The water H atoms were located and refined with the distance restraint O—H = 0.85 (1) Å.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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