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catena-Poly[[tetrakis(μ-acetato-κO:$\kappa O'$)dicopper(II)]-μ-2-aminopyrimidine-$\kappa^2 N$:N'] at 150 K

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Key indicators

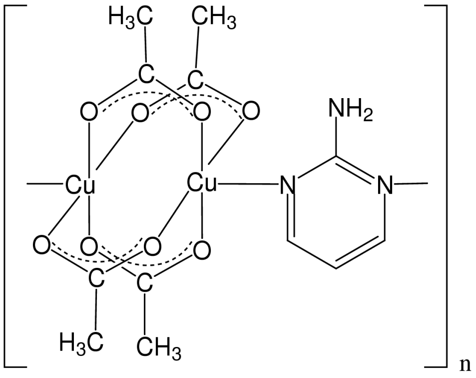
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.024
wR factor = 0.064
Data-to-parameter ratio = 12.6

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

The basic architectural motif of the title compound, $[Cu_2(C_2H_3O_2)_4(C_4H_5N_3)]$, is a chain of alternating tetrakis(μ-acetato)dicopper(II) and 2-aminopyrimidine moieties in which square-pyramidal copper(II) centres are alternately linked by four bridging acetate anions [Cu—O = 1.947 (2)–1.984 (2) Å] and a single bridging 2-aminopyrimidine molecule [Cu—N 2.214 (2) Å]. N—H···O and C—H···O hydrogen bonds support the Cu—N coordination to generate the chains, which align perpendicular to the $(10\overline{1})$ plane. The dinuclear copper unit has crystallographic inversion symmetry, and the 2-aminopyrimidine bridging ligand has twofold rotation symmetry.

Comment

The structure of the title compound, (I), has been reported at 296 K (Smith *et al.*, 1991). Continuing our studies of the coordination chemistry of the tetrakis(acetato)dicopper(II) moiety with nitrogen bases (Begley *et al.*, 1993), we now report the structure of the title compound at 150 K. Its asymmetric unit comprises half a tetrakis(acetato)dicopper(II) moiety, the complete dinuclear unit being located on an inversion centre, and half a 2-aminopyrimidine ligand, which is located on a twofold symmetry axis passing through N12, C12 and C14 (Fig. 1). The square-pyramidal coordination geometry of the copper(II) centre comprises four basal O atoms from four bridging acetate anions [Cu—O 1.947 (2), 1.948 (2), 1.983 (2) and, 1.984(2) Å] and an axial N atom from a single bridging 2-aminopyrimidine molecule [Cu—N 2.214 (2) Å].



(I)

The basic architectural motif of the extended structure is a zigzag chain (Fig. 2) of alternating tetrakis(acetato)dicopper(II) and 2-aminopyrimidine moieties. The zigzag motif of the chains, which pack perpendicular to the $(10\overline{1})$

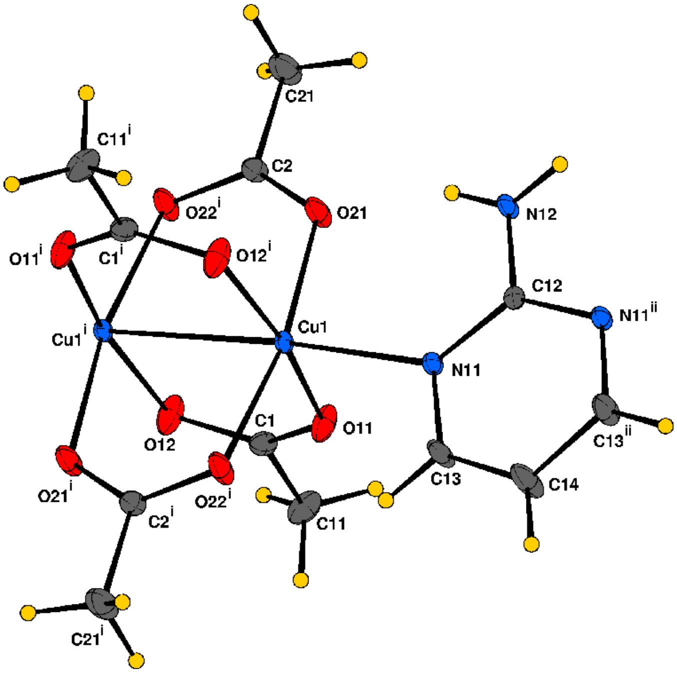


Figure 1
A view of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry codes: (i) $1/2-x$, $1/2-y$, $1-z$; (ii) $-x$, y, $3/2-z$.]

plane (Fig. 3) with alternating copper(II)–copper(II) separations of 2.6281 (14) and 6.4589 (14) Å, results from the 120° angle between the σ-donor orbitals of the pyrimidine heterocyclic N atoms. Two hydrogen-bonding contacts, one N—H···O and one C—H···O, support the Cu—N coordination to generate the chain structure. The significance of the hydrogen-bonding interactions comes, not from their dimensions [N—H 0.86 (2), H···O 2.04 (2), N···O 2.872 (3) Å and N—H···O 161 (3)°; C—H 0.93, H···O 2.34, C···O 2.987 (3) Å and C—H···O 127°], which are moderated by the Cu—N coordination, but from the low value of the dihedral angle (7.2°) between the plane containing the copper centres and the pyrimidine molecule and the plane containing the copper centres and the two acetate anions providing the hydrogen-bond acceptors.

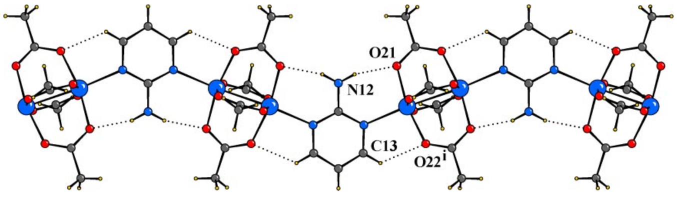


Figure 2
A view of the zigzag chain of alternating tetrakis(acetato)dicopper(II) and 2-aminopyrimidine moieties. Atoms are identified as follows: Cu, large blue circles; C, black circles; N, blue circles; O, red circles; H, small yellow circles. [Symmetry code: (i) $1/2-x$, $1/2-y$, $1-z$.]

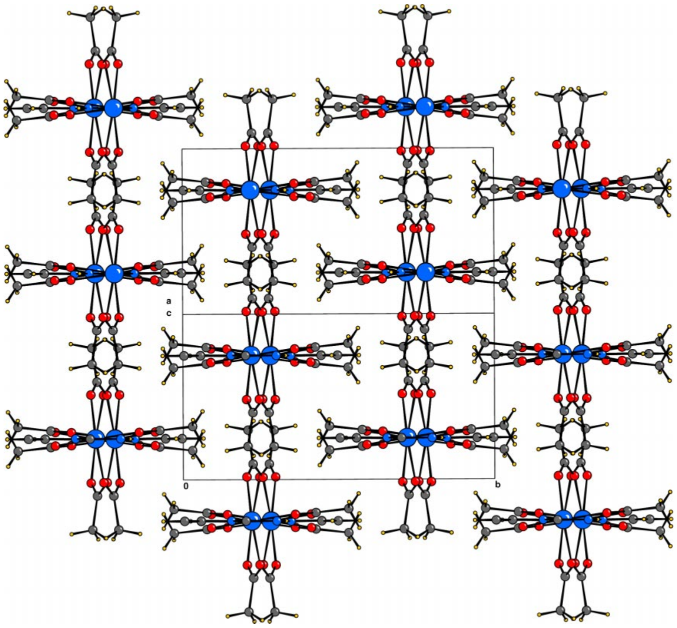


Figure 3
A view of the extended structure of the title compound perpendicular to the $(10\overline{1})$ plane, showing the packing of the chains. Atoms are identified as in Fig. 2.

Experimental

Careful layering of a methanol solution (5 ml) of $Cu(OAc)_2 \cdot H_2O$ (0.079 g, 0.396 mmol) with a dichloromethane solution (5 ml) of *N,N'*-bis(pyrimidin-2-yl)thiourea (0.090 g, 0.387 mmol), previously prepared by reaction of 2-aminopyrimidine (2.02 g, 21.2 mol) with carbon disulfide (2.41 g, 31.7 mmol) and sulfur (0.178 g, 5.55 mmol) in ethanol (50 ml), yielded, over a period of 7 d, two sets of large chunky crystals, which proved to be $Cu(OAc)_2 \cdot H_2O$ (located at the bottom of the vial) and the title compound (located on the walls of the vial). The latter was assumed to arise *via* ligand decomposition.

Crystal data

$[Cu_2(C_2H_3O_2)_4(C_4H_5N_3)]$
$M_r = 458.36$
Monoclinic, $C2/c$
$a = 15.096$ (8) Å
$b = 13.585$ (8) Å
$c = 8.580$ (4) Å
$\beta = 94.13$ (5)°
$V = 1755.0$ (16) Å^3
$Z = 4$
$D_x = 1.735$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 12.8$–17.0°
$\mu = 2.47$ mm^{-1}
$T = 150$ (2) K
Tablet, dark green
0.53 × 0.45 × 0.20 mm

Data collection

Stoe Stadi-4 four-circle diffractometer
ω/θ scans
Absorption correction: numerical (*X-RED*; Stoe & Cie, 1995)
$T_{min} = 0.375$, $T_{max} = 0.623$
1980 measured reflections
1517 independent reflections
1428 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 25.0$°
$h = -17 \rightarrow 17$
$k = 0 \rightarrow 16$
$l = -2 \rightarrow 10$
3 standard reflections
frequency: 60 min
intensity decay: none

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Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 4.087P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.024$	$(\Delta/\sigma)_{\max} = 0.001$
$wR(F^2) = 0.064$	$\Delta\rho_{\max} = 0.32$ e Å^{-3}
$S = 1.15$	$\Delta\rho_{\min} = -0.45$ e Å^{-3}
1517 reflections	
120 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Selected interatomic distances (Å).

Cu1—Cu1^{i}	2.6281 (14)	Cu1—O12^{i}	1.947 (2)
Cu1—N11	2.214 (2)	Cu1—O21	1.984 (2)
Cu1—O11	1.949 (2)	Cu1—O22^{i}	1.983 (2)

Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$.

Amino H atoms were located from ΔF syntheses and refined with the N—H distance restrained to be 0.88 (1) Å and with $U_{\rm iso}(H) = 1.5U_{\rm eq}(N)$. Methyl H atoms were located from ΔF syntheses and refined as part of a rigid rotating group with C—H = 0.96 Å and with $U_{\rm iso}(H) = 1.5U_{\rm eq}(C)$. After location from ΔF syntheses, aromatic H atoms were placed geometrically and refined with a riding model for which the C—H distance was constrained to be 0.93 Å and $U_{\rm iso}(H) = 1.2U_{\rm eq}(C)$.

Data collection: *Stadi*4 (Stoe & Cie, 1995); cell refinement: *Stadi*4; data reduction: *X-RED* (Stoe & Cie, 1995); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2002).

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