

Tetra- μ -acetato- $\kappa^8 O:O'$ -bis[(2-amino-3,5-dichloropyridine- κN^1)copper(II)]-(Cu—Cu)

Hui-Chang Chang, Jacqueline M. Cole,*†Tze-Chia Lin and Paul G. Waddell‡

Cavendish Laboratory, University of Cambridge, J. J. Thomson Avenue, Cambridge CB3 0HE, England

Correspondence e-mail: jmc61@cam.ac.uk

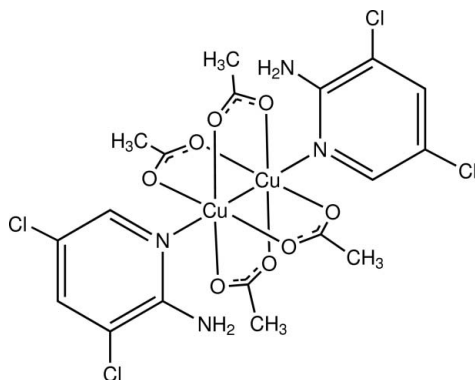
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Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}—\text{C}) = 0.003$ Å; R factor = 0.029; wR factor = 0.077; data-to-parameter ratio = 18.2.

The title binuclear Cu(II) complex, $[\text{Cu}_2(\text{CH}_3\text{CO}_2)_4(\text{C}_5\text{H}_4\text{Cl}_2\text{N}_2)_2]$, is disposed about a crystallographic inversion center, located at the mid-point of the Cu—Cu connecting line. The Cu...Cu distance is 2.6600 (6) Å and each metal atom exhibits a Jahn–Teller-distorted octahedral geometry.

Related literature

For the structures of polymorphic tetrakis(μ -acetato- $O:O'$)bis[(pyridine- N)copper(II)], see: Barclay & Kennard (1961); Hanic *et al.* (1964); Uekusa *et al.* (1989).



Experimental

Crystal data

$[\text{Cu}_2(\text{C}_2\text{H}_3\text{O}_2)_4(\text{C}_5\text{H}_4\text{Cl}_2\text{N}_2)_2]$	$V = 1279.0$ (4) Å ³
$M_r = 689.26$	$Z = 2$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 8.2857$ (17) Å	$\mu = 2.13$ mm ^{−1}
$b = 17.010$ (3) Å	$T = 150$ K
$c = 9.3159$ (19) Å	$0.44 \times 0.37 \times 0.17$ mm
$\beta = 103.07$ (3)°	

Data collection

Rigaku Saturn724+ diffractometer	18645 measured reflections
Absorption correction: multi-scan (<i>ABSCOR</i> ; Higashi, 1995)	3028 independent reflections
$T_{\min} = 0.407$, $T_{\max} = 0.696$	2996 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$	166 parameters
$wR(F^2) = 0.077$	H-atom parameters constrained
$S = 1.10$	$\Delta\rho_{\max} = 0.51$ e Å ^{−3}
3028 reflections	$\Delta\rho_{\min} = -0.35$ e Å ^{−3}

Data collection: *CrystalClear* (Rigaku/MS, 2008); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2343).

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† Other affiliation: Department of Chemistry, University of New Brunswick, Fredericton, NB, Canada E3B 5A3.

supporting information

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Tetra- μ -acetato- κ^8 O:O'-bis[(2-amino-3,5-dichloropyridine- κ N¹)copper(II)](Cu—Cu)

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S1. Comment

The title compound is binuclear and disposed about a crystallographic centre of symmetry with a Cu—Cu distance of 2.6600 (8) Å. It has a similar geometry to that observed in the two known polymorphs of monopyridinecopper(II) acetate (Barclay & Kennard, 1961; Hanic *et al.*, 1964; Uekusa *et al.*, 1989). However, the Cu—N bond distance in the title compound is *ca.* 0.05 Å longer than that observed in the orthorhombic polymorph and *ca.* 0.08 Å longer than that in the monoclinic polymorph.

S2. Experimental

A suspension of (3,5-dichloro-2-pyridylimino)-*o*-cresol copper (II) (1 mg, 0.0016 mmol) in ethanol (*ca.* 3 ml) was heated to *ca.* 323 K until fully dissolved. The solution was then allowed to cool to room temperature. Crystals suitable for single-crystal X-ray crystallography were grown *via* slow evaporation of methanol over seven days.

S3. Refinement

All H atoms were placed in idealized positions and refined as riding to their parent atoms, with bond lengths fixed to C—H = 0.93 (aromatic CH), 0.96 (methyl CH₃) or 0.86 Å (amine NH₂). Isotropic displacement parameters were calculated as $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{carrier atom})$ for methyl groups and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{carrier atom})$ otherwise.

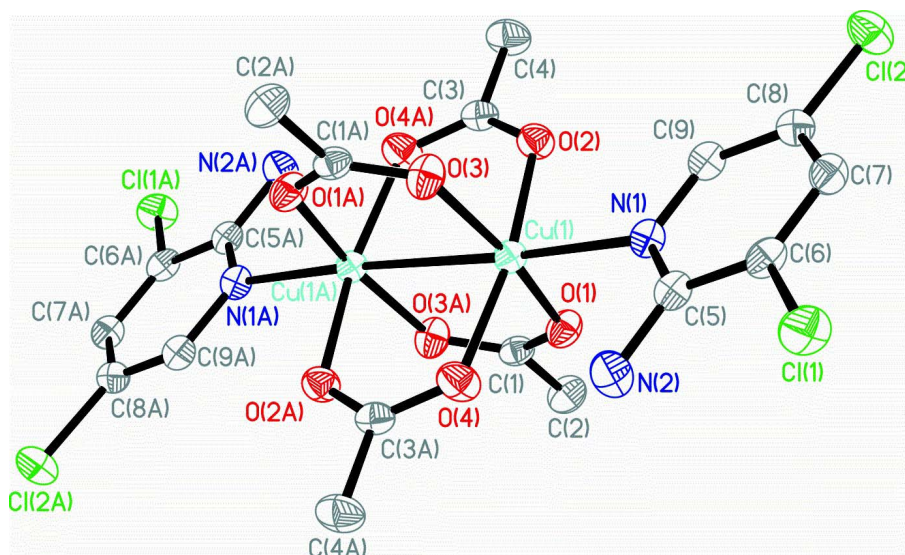


Figure 1

The structure of the title compound with displacement ellipsoids drawn at the 50% probability level; hydrogen atoms omitted for clarity. Atoms labeled with flag A are generated by symmetry 1-x, 1-y, 1-z.

Tetra- μ -acetato- κ^8 O':O'-bis[(2-amino-3,5-dichloropyridine- κN^1)copper(II)](Cu—Cu)

Crystal data

$[\text{Cu}_2(\text{C}_2\text{H}_3\text{O}_2)_4(\text{C}_5\text{H}_4\text{Cl}_2\text{N}_2)_2]$

$M_r = 689.26$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.2857(17) \text{ \AA}$

$b = 17.010(3) \text{ \AA}$

$c = 9.3159(19) \text{ \AA}$

$\beta = 103.07(3)^\circ$

$V = 1279.0(4) \text{ \AA}^3$

$Z = 2$

$F(000) = 692$

$D_x = 1.79 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 6896 reflections

$\theta = 4.8\text{--}36.7^\circ$

$\mu = 2.13 \text{ mm}^{-1}$

$T = 150 \text{ K}$

Prism, blue

$0.44 \times 0.37 \times 0.17 \text{ mm}$

Data collection

Rigaku Saturn724+
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: $28.5714 \text{ pixels mm}^{-1}$

ω scans

Absorption correction: multi-scan

(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.407$, $T_{\max} = 0.696$

18645 measured reflections

3028 independent reflections

2996 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.032$

$\theta_{\max} = 28.3^\circ$, $\theta_{\min} = 4.8^\circ$

$h = -11 \rightarrow 11$

$k = -22 \rightarrow 20$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.077$

$S = 1.10$

3028 reflections

166 parameters

0 restraints

0 constraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 0.9376P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.51 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.35 \text{ e } \text{\AA}^{-3}$$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu1	0.42778 (2)	0.430189 (12)	0.47068 (2)	0.01965 (8)
Cl1	0.39574 (7)	0.08106 (3)	0.48552 (6)	0.03401 (12)
Cl2	−0.03256 (6)	0.24404 (3)	0.07032 (6)	0.03440 (12)
N2	0.5053 (2)	0.24052 (10)	0.5958 (2)	0.0307 (4)
H10	0.5454	0.2843	0.6342	0.037*
H8	0.5429	0.1967	0.6361	0.037*
O1	0.26762 (16)	0.47238 (8)	0.57766 (15)	0.0273 (3)
N1	0.32520 (17)	0.30954 (9)	0.40958 (17)	0.0219 (3)
O3	0.60795 (16)	0.41098 (8)	0.36793 (17)	0.0288 (3)
C7	0.1925 (2)	0.16842 (10)	0.2815 (2)	0.0247 (3)
H7	0.1496	0.1214	0.2379	0.03*
C3	0.3140 (2)	0.55353 (11)	0.26647 (19)	0.0232 (3)
C1	0.2771 (2)	0.54062 (11)	0.63179 (19)	0.0225 (3)
C8	0.1324 (2)	0.24062 (11)	0.2229 (2)	0.0235 (3)
C9	0.2019 (2)	0.30880 (10)	0.2871 (2)	0.0234 (3)
H9	0.1624	0.3565	0.2445	0.028*
C5	0.3835 (2)	0.24049 (10)	0.4722 (2)	0.0226 (3)
O4	0.57808 (18)	0.40136 (8)	0.65883 (16)	0.0313 (3)
C6	0.3169 (2)	0.16920 (10)	0.4057 (2)	0.0234 (3)
C4	0.1985 (3)	0.58896 (14)	0.1340 (2)	0.0343 (4)
H5	0.2616	0.6087	0.0672	0.052*
H6	0.1377	0.6313	0.165	0.052*
H4	0.1226	0.5495	0.0855	0.052*
O2	0.29538 (16)	0.48257 (8)	0.29448 (14)	0.0268 (3)
C2	0.1372 (2)	0.56626 (12)	0.7009 (2)	0.0311 (4)
H2	0.1617	0.6172	0.7448	0.047*
H1	0.1249	0.5291	0.7752	0.047*
H3	0.0361	0.5688	0.6264	0.047*

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.02168 (12)	0.01600 (12)	0.02151 (13)	0.00133 (6)	0.00540 (8)	0.00034 (7)
Cl1	0.0456 (3)	0.0184 (2)	0.0364 (3)	0.00489 (17)	0.0057 (2)	0.00646 (17)
Cl2	0.0360 (2)	0.0319 (3)	0.0301 (2)	0.00245 (18)	−0.00326 (19)	−0.00169 (18)
N2	0.0313 (8)	0.0209 (8)	0.0354 (9)	0.0017 (6)	−0.0016 (7)	0.0037 (6)
O1	0.0303 (6)	0.0243 (6)	0.0310 (7)	−0.0009 (5)	0.0144 (5)	−0.0015 (5)
N1	0.0219 (6)	0.0182 (7)	0.0267 (7)	0.0017 (5)	0.0077 (5)	0.0000 (6)
O3	0.0269 (6)	0.0249 (6)	0.0377 (7)	0.0009 (5)	0.0139 (5)	−0.0034 (6)
C7	0.0291 (8)	0.0191 (8)	0.0276 (9)	−0.0010 (6)	0.0101 (7)	−0.0033 (7)

C3	0.0244 (8)	0.0254 (8)	0.0203 (8)	0.0064 (6)	0.0059 (6)	0.0020 (6)
C1	0.0238 (8)	0.0237 (8)	0.0206 (7)	0.0049 (6)	0.0063 (6)	0.0043 (6)
C8	0.0236 (8)	0.0238 (8)	0.0234 (8)	0.0025 (6)	0.0060 (6)	0.0001 (6)
C9	0.0233 (7)	0.0203 (8)	0.0277 (8)	0.0034 (6)	0.0084 (6)	0.0016 (7)
C5	0.0226 (7)	0.0202 (8)	0.0267 (9)	0.0023 (6)	0.0091 (6)	0.0012 (6)
O4	0.0382 (7)	0.0223 (7)	0.0289 (7)	0.0014 (5)	−0.0019 (5)	0.0038 (5)
C6	0.0285 (8)	0.0156 (7)	0.0283 (9)	0.0039 (6)	0.0112 (7)	0.0037 (6)
C4	0.0334 (10)	0.0389 (11)	0.0278 (9)	0.0106 (8)	0.0009 (7)	0.0072 (8)
O2	0.0295 (6)	0.0239 (6)	0.0250 (6)	0.0007 (5)	0.0023 (5)	0.0022 (5)
C2	0.0301 (9)	0.0328 (10)	0.0346 (10)	0.0050 (7)	0.0159 (8)	−0.0004 (8)

Geometric parameters (Å, °)

Cu1—O1	1.9665 (14)	C7—H7	0.93
Cu1—O4	1.9688 (15)	C3—O2	1.252 (2)
Cu1—O2	1.9691 (14)	C3—O4 ⁱ	1.262 (2)
Cu1—O3	1.9743 (14)	C3—C4	1.505 (2)
Cu1—N1	2.2449 (15)	C1—O3 ⁱ	1.259 (2)
Cu1—Cu1 ⁱ	2.6600 (6)	C1—C2	1.511 (2)
Cl1—C6	1.7342 (18)	C8—C9	1.371 (3)
Cl2—C8	1.735 (2)	C9—H9	0.93
N2—C5	1.349 (2)	C5—C6	1.416 (3)
N2—H10	0.86	O4—C3 ⁱ	1.262 (2)
N2—H8	0.86	C4—H5	0.96
O1—C1	1.261 (2)	C4—H6	0.96
N1—C9	1.348 (2)	C4—H4	0.96
N1—C5	1.351 (2)	C2—H2	0.96
O3—C1 ⁱ	1.259 (2)	C2—H1	0.96
C7—C6	1.365 (3)	C2—H3	0.96
C7—C8	1.390 (2)		
O1—Cu1—O4	90.18 (6)	O3 ⁱ —C1—O1	125.55 (16)
O1—Cu1—O2	86.83 (6)	O3 ⁱ —C1—C2	117.59 (17)
O4—Cu1—O2	167.37 (6)	O1—C1—C2	116.85 (16)
O1—Cu1—O3	167.68 (6)	C9—C8—C7	119.85 (17)
O4—Cu1—O3	89.71 (7)	C9—C8—Cl2	120.30 (14)
O2—Cu1—O3	90.60 (6)	C7—C8—Cl2	119.83 (14)
O1—Cu1—N1	101.65 (6)	N1—C9—C8	122.72 (16)
O4—Cu1—N1	97.18 (6)	N1—C9—H9	118.6
O2—Cu1—N1	95.43 (6)	C8—C9—H9	118.6
O3—Cu1—N1	90.58 (6)	N2—C5—N1	119.58 (16)
O1—Cu1—Cu1 ⁱ	83.91 (4)	N2—C5—C6	121.08 (16)
O4—Cu1—Cu1 ⁱ	82.81 (5)	N1—C5—C6	119.34 (16)
O2—Cu1—Cu1 ⁱ	84.67 (4)	C3 ⁱ —O4—Cu1	124.50 (12)
O3—Cu1—Cu1 ⁱ	83.85 (4)	C7—C6—C5	121.61 (16)
N1—Cu1—Cu1 ⁱ	174.44 (4)	C7—C6—Cl1	119.62 (14)
C5—N2—H10	120	C5—C6—Cl1	118.77 (14)
C5—N2—H8	120	C3—C4—H5	109.5

H10—N2—H8	120	C3—C4—H6	109.5
C1—O1—Cu1	123.41 (12)	H5—C4—H6	109.5
C9—N1—C5	119.05 (15)	C3—C4—H4	109.5
C9—N1—Cu1	113.20 (11)	H5—C4—H4	109.5
C5—N1—Cu1	127.40 (12)	H6—C4—H4	109.5
C1 ⁱ —O3—Cu1	123.07 (12)	C3—O2—Cu1	122.58 (12)
C6—C7—C8	117.37 (16)	C1—C2—H2	109.5
C6—C7—H7	121.3	C1—C2—H1	109.5
C8—C7—H7	121.3	H2—C2—H1	109.5
O2—C3—O4 ⁱ	125.23 (16)	C1—C2—H3	109.5
O2—C3—C4	118.13 (17)	H2—C2—H3	109.5
O4 ⁱ —C3—C4	116.64 (17)	H1—C2—H3	109.5

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