

## Tetra- $\mu$ -acetato- $\kappa^8$ O:O'-bis{[2-(*m*-tolyl-amino)pyridine- $\kappa$ N]copper(II)}

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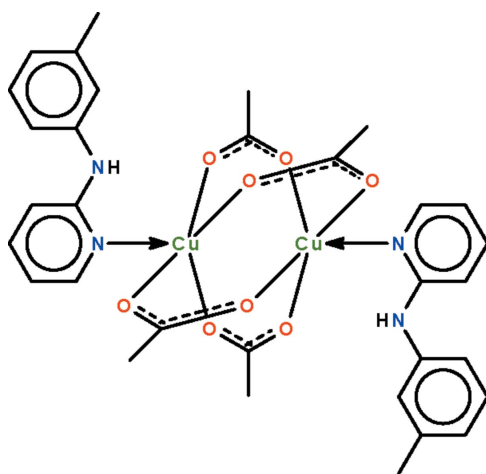
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Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.038;  $wR$  factor = 0.098; data-to-parameter ratio = 17.4.

In the crystal structure of the title compound,  $[\text{Cu}_2(\text{C}_2\text{H}_3\text{O}_2)_4(\text{C}_{12}\text{H}_{12}\text{N}_2)_2]$ , the binuclear molecule lies about a center of inversion; the four acetate groups each bridge a pair of  $\text{Cu}^{\text{II}}$  atoms. The coordination of the metal atom is distorted square-pyramidal, with the bonding O atoms comprising a square basal plane and the coordinating N atom of the *N*-heterocycle occupying the apical position. The pyridine ring is twisted with respect to the benzene ring at a dihedral angle of  $45.68$  (16)°. Intramolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonding is present between the imino and carboxy groups.

### Related literature

There are many examples of tetrakisacetatobis(substituted pyridine)copper complexes. For examples of 2-aminopyridyl derivatives, see: Barquín *et al.* (2004); Seco *et al.* (2004); Sieroń (2004).



### Experimental

#### Crystal data

$[\text{Cu}_2(\text{C}_2\text{H}_3\text{O}_2)_4(\text{C}_{12}\text{H}_{12}\text{N}_2)_2]$	$\gamma = 78.568$ (2)°
$M_r = 731.73$	$V = 823.51$ (4) Å <sup>3</sup>
Triclinic, $P\bar{1}$	$Z = 1$
$a = 7.7143$ (2) Å	Mo $K\alpha$ radiation
$b = 10.5625$ (3) Å	$\mu = 1.35$ mm <sup>-1</sup>
$c = 11.2413$ (3) Å	$T = 293$ K
$\alpha = 66.531$ (2)°	$0.25 \times 0.15 \times 0.05$ mm
$\beta = 85.740$ (2)°	

#### Data collection

Bruker SMART APEX diffractometer	6451 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	3678 independent reflections
$T_{\text{min}} = 0.730$ , $T_{\text{max}} = 0.936$	2915 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.023$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$	211 parameters
$wR(F^2) = 0.098$	H-atom parameters constrained
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.49$ e Å <sup>-3</sup>
3678 reflections	$\Delta\rho_{\text{min}} = -0.59$ e Å <sup>-3</sup>

**Table 1**

Selected bond lengths (Å).

Cu1—O1	1.9762 (19)	Cu1—O4 <sup>i</sup>	1.966 (2)
Cu1—O2 <sup>i</sup>	1.9866 (19)	Cu1—N1	2.197 (2)
Cu1—O3	1.967 (2)		

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

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2}\cdots\text{O2}^i$	0.86	2.17	2.913 (3)	145

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *pubCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2688).

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**supplementary materials**

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## Tetra- $\mu$ -acetato- $\kappa^8 O:O'$ -bis{[2-(*m*-tolylamino)pyridine- $\kappa N$ ]copper(II)}

Z. A. Fairuz, Z. Aiyub, Z. Abdullah and S. W. Ng

### Experimental

Copper acetate (0.1 g, 0.5 mmol) was dissolved in acetonitrile (5 ml). The solution was mixed with a solution of 3-tolylamino-2-pyridine (0.2 g, 1.1 mmol) dissolved in acetonitrile (15 ml). The green precipitate that formed was recrystallized from acetonitrile.

### Refinement

Carbon-bound H-atoms were placed in calculated positions (C–H 0.93–0.96 Å) and were included in the refinement in the riding model approximation, with  $U(H)$  set to 1.2–1.5 $U(C)$ . The amino H-atom was similarly treated.

### Figures

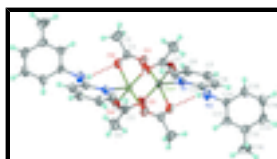


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of  $\text{Cu}_2(\text{C}_2\text{H}_3\text{O}_2)_4(\text{C}_{12}\text{H}_{12}\text{N}_2)_2$  at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. Dashed lines indicate the hydrogen bonding.

## Tetra- $\mu$ -acetato- $\kappa^8 O:O'$ -bis{[2-(*m*-tolylamino)pyridine- $\kappa N$ ]copper(II)}

### Crystal data

$[\text{Cu}_2(\text{C}_2\text{H}_3\text{O}_2)_4(\text{C}_{12}\text{H}_{12}\text{N}_2)_2]$	$Z = 1$
$M_r = 731.73$	$F(000) = 378$
Triclinic, $P\bar{1}$	$D_x = 1.475 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 7.7143 (2) \text{ \AA}$	Cell parameters from 2561 reflections
$b = 10.5625 (3) \text{ \AA}$	$\theta = 2.3\text{--}27.6^\circ$
$c = 11.2413 (3) \text{ \AA}$	$\mu = 1.35 \text{ mm}^{-1}$
$\alpha = 66.531 (2)^\circ$	$T = 293 \text{ K}$
$\beta = 85.740 (2)^\circ$	Prism, green
$\gamma = 78.568 (2)^\circ$	$0.25 \times 0.15 \times 0.05 \text{ mm}$
$V = 823.51 (4) \text{ \AA}^3$	

### Data collection

Bruker SMART APEX diffractometer	3678 independent reflections
Radiation source: fine-focus sealed tube	2915 reflections with $I > 2\sigma(I)$

# supplementary materials

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graphite  $R_{\text{int}} = 0.023$   
 $\omega$  scans  $\theta_{\text{max}} = 27.5^\circ$ ,  $\theta_{\text{min}} = 2.0^\circ$   
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996)  $h = -10 \rightarrow 9$   
 $T_{\text{min}} = 0.730$ ,  $T_{\text{max}} = 0.936$   $k = -13 \rightarrow 13$   
6451 measured reflections  $l = -14 \rightarrow 14$

## Refinement

Refinement on  $F^2$  Primary atom site location: structure-invariant direct methods  
Least-squares matrix: full Secondary atom site location: difference Fourier map  
 $R[F^2 > 2\sigma(F^2)] = 0.038$  Hydrogen site location: inferred from neighbouring sites  
 $wR(F^2) = 0.098$  H-atom parameters constrained  
 $S = 1.07$   $w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.4239P]$   
3678 reflections where  $P = (F_o^2 + 2F_c^2)/3$   
211 parameters  $(\Delta/\sigma)_{\text{max}} = 0.001$   
0 restraints  $\Delta\rho_{\text{max}} = 0.49 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.59 \text{ e } \text{\AA}^{-3}$

## 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	0.58894 (4)	0.46743 (4)	0.60797 (3)	0.03044 (12)
O1	0.6955 (3)	0.6324 (2)	0.50147 (18)	0.0439 (5)
O2	0.5505 (3)	0.6857 (2)	0.31997 (18)	0.0419 (5)
O3	0.7582 (3)	0.3494 (2)	0.53769 (19)	0.0460 (5)
O4	0.6100 (3)	0.4052 (2)	0.35536 (19)	0.0436 (5)
N1	0.7506 (3)	0.4120 (2)	0.7806 (2)	0.0304 (5)
N2	0.5409 (3)	0.3175 (3)	0.9254 (2)	0.0446 (6)
H2	0.4771	0.3436	0.8573	0.053*
C1	0.6579 (4)	0.7076 (3)	0.3856 (3)	0.0342 (6)
C2	0.7467 (5)	0.8317 (4)	0.3197 (3)	0.0570 (9)
H2A	0.8448	0.8237	0.3717	0.085*
H2B	0.6637	0.9162	0.3091	0.085*
H2C	0.7885	0.8346	0.2363	0.085*
C3	0.7354 (4)	0.3375 (3)	0.4329 (3)	0.0378 (6)
C4	0.8709 (5)	0.2335 (4)	0.4000 (3)	0.0578 (9)
H4A	0.9175	0.1573	0.4784	0.087*
H4B	0.9652	0.2787	0.3520	0.087*
H4C	0.8169	0.1979	0.3485	0.087*
C5	0.9143 (4)	0.4436 (3)	0.7539 (3)	0.0389 (7)
H5	0.9471	0.4835	0.6675	0.047*
C6	1.0348 (4)	0.4198 (3)	0.8479 (3)	0.0438 (7)
H6	1.1454	0.4446	0.8259	0.053*
C7	0.9871 (4)	0.3582 (3)	0.9752 (3)	0.0403 (7)
H7	1.0662	0.3400	1.0411	0.048*

C8	0.8228 (4)	0.3236 (3)	1.0051 (3)	0.0395 (7)
H8	0.7896	0.2817	1.0912	0.047*
C9	0.7059 (3)	0.3518 (3)	0.9053 (2)	0.0309 (6)
C10	0.4629 (4)	0.2444 (3)	1.0447 (2)	0.0339 (6)
C11	0.3760 (4)	0.1388 (3)	1.0512 (3)	0.0365 (6)
H11	0.3785	0.1149	0.9799	0.044*
C12	0.2858 (4)	0.0682 (3)	1.1613 (3)	0.0417 (7)
C13	0.2886 (5)	0.1024 (4)	1.2678 (3)	0.0513 (8)
H13	0.2327	0.0539	1.3440	0.062*
C14	0.3736 (5)	0.2078 (4)	1.2618 (3)	0.0522 (8)
H14	0.3734	0.2302	1.3339	0.063*
C15	0.4592 (4)	0.2808 (3)	1.1506 (3)	0.0420 (7)
H15	0.5137	0.3533	1.1468	0.050*
C16	0.1823 (5)	-0.0390 (4)	1.1628 (4)	0.0658 (10)
H16A	0.2210	-0.0685	1.0932	0.099*
H16B	0.0587	0.0016	1.1522	0.099*
H16C	0.2012	-0.1187	1.2440	0.099*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cu1	0.02917 (19)	0.0399 (2)	0.02439 (17)	-0.01136 (14)	0.00032 (12)	-0.01252 (14)
O1	0.0492 (13)	0.0523 (13)	0.0312 (10)	-0.0263 (10)	-0.0027 (9)	-0.0090 (9)
O2	0.0452 (12)	0.0516 (13)	0.0325 (10)	-0.0246 (10)	-0.0018 (9)	-0.0125 (9)
O3	0.0414 (12)	0.0601 (14)	0.0384 (11)	-0.0004 (10)	-0.0021 (9)	-0.0250 (10)
O4	0.0412 (12)	0.0558 (13)	0.0376 (11)	-0.0023 (10)	-0.0020 (9)	-0.0251 (10)
N1	0.0282 (12)	0.0365 (13)	0.0277 (11)	-0.0101 (10)	-0.0003 (9)	-0.0118 (10)
N2	0.0374 (14)	0.0714 (18)	0.0249 (11)	-0.0241 (13)	-0.0002 (10)	-0.0123 (12)
C1	0.0330 (15)	0.0411 (16)	0.0309 (14)	-0.0141 (12)	0.0046 (11)	-0.0138 (12)
C2	0.065 (2)	0.058 (2)	0.0484 (19)	-0.0362 (19)	-0.0014 (16)	-0.0094 (16)
C3	0.0370 (16)	0.0418 (17)	0.0353 (15)	-0.0110 (13)	0.0082 (12)	-0.0154 (13)
C4	0.058 (2)	0.062 (2)	0.051 (2)	0.0063 (18)	0.0032 (16)	-0.0289 (18)
C5	0.0349 (16)	0.0487 (18)	0.0323 (14)	-0.0123 (14)	0.0022 (12)	-0.0133 (13)
C6	0.0298 (15)	0.0532 (19)	0.0489 (18)	-0.0094 (14)	-0.0038 (13)	-0.0191 (15)
C7	0.0344 (16)	0.0476 (18)	0.0387 (16)	-0.0007 (13)	-0.0117 (12)	-0.0179 (14)
C8	0.0398 (17)	0.0489 (18)	0.0277 (14)	-0.0074 (14)	-0.0039 (12)	-0.0125 (13)
C9	0.0293 (14)	0.0353 (15)	0.0289 (13)	-0.0068 (11)	-0.0014 (10)	-0.0129 (11)
C10	0.0299 (14)	0.0421 (16)	0.0272 (13)	-0.0056 (12)	0.0013 (10)	-0.0117 (12)
C11	0.0327 (15)	0.0406 (16)	0.0362 (15)	-0.0027 (12)	0.0003 (11)	-0.0171 (13)
C12	0.0366 (16)	0.0317 (16)	0.0494 (18)	-0.0040 (13)	0.0041 (13)	-0.0100 (13)
C13	0.052 (2)	0.055 (2)	0.0358 (16)	-0.0099 (16)	0.0131 (14)	-0.0090 (15)
C14	0.058 (2)	0.068 (2)	0.0357 (16)	-0.0142 (18)	0.0102 (14)	-0.0261 (16)
C15	0.0450 (18)	0.0476 (18)	0.0385 (16)	-0.0129 (15)	0.0054 (13)	-0.0210 (14)
C16	0.065 (3)	0.047 (2)	0.080 (3)	-0.0220 (19)	0.006 (2)	-0.0163 (19)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Cu1—O1	1.9762 (19)	C4—H4C	0.9600
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## supplementary materials

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Cu1—O2 <sup>i</sup>	1.9866 (19)	C5—C6	1.372 (4)
Cu1—O3	1.967 (2)	C5—H5	0.9300
Cu1—O4 <sup>i</sup>	1.966 (2)	C6—C7	1.372 (4)
Cu1—N1	2.197 (2)	C6—H6	0.9300
Cu1—Cu1 <sup>i</sup>	2.6532 (6)	C7—C8	1.370 (4)
O1—C1	1.246 (3)	C7—H7	0.9300
O2—C1	1.259 (3)	C8—C9	1.393 (4)
O2—Cu1 <sup>i</sup>	1.9866 (19)	C8—H8	0.9300
O3—C3	1.261 (3)	C10—C11	1.387 (4)
O4—C3	1.250 (4)	C10—C15	1.385 (4)
O4—Cu1 <sup>i</sup>	1.966 (2)	C11—C12	1.384 (4)
N1—C9	1.339 (3)	C11—H11	0.9300
N1—C5	1.352 (3)	C12—C13	1.385 (4)
N2—C9	1.370 (3)	C12—C16	1.503 (4)
N2—C10	1.413 (3)	C13—C14	1.377 (5)
N2—H2	0.8600	C13—H13	0.9300
C1—C2	1.498 (4)	C14—C15	1.380 (4)
C2—H2A	0.9600	C14—H14	0.9300
C2—H2B	0.9600	C15—H15	0.9300
C2—H2C	0.9600	C16—H16A	0.9600
C3—C4	1.500 (4)	C16—H16B	0.9600
C4—H4A	0.9600	C16—H16C	0.9600
C4—H4B	0.9600		
O4 <sup>i</sup> —Cu1—O3	167.64 (8)	H4A—C4—H4C	109.5
O4 <sup>i</sup> —Cu1—O1	88.77 (9)	H4B—C4—H4C	109.5
O3—Cu1—O1	90.33 (9)	N1—C5—C6	123.4 (3)
O4 <sup>i</sup> —Cu1—O2 <sup>i</sup>	89.16 (9)	N1—C5—H5	118.3
O3—Cu1—O2 <sup>i</sup>	89.01 (9)	C6—C5—H5	118.3
O1—Cu1—O2 <sup>i</sup>	167.27 (8)	C5—C6—C7	118.1 (3)
O4 <sup>i</sup> —Cu1—N1	98.40 (8)	C5—C6—H6	121.0
O3—Cu1—N1	93.96 (8)	C7—C6—H6	121.0
O1—Cu1—N1	94.60 (8)	C8—C7—C6	119.8 (3)
O2 <sup>i</sup> —Cu1—N1	98.13 (8)	C8—C7—H7	120.1
O4 <sup>i</sup> —Cu1—Cu1 <sup>i</sup>	84.47 (6)	C6—C7—H7	120.1
O3—Cu1—Cu1 <sup>i</sup>	83.19 (6)	C7—C8—C9	119.3 (3)
O1—Cu1—Cu1 <sup>i</sup>	83.69 (6)	C7—C8—H8	120.3
O2 <sup>i</sup> —Cu1—Cu1 <sup>i</sup>	83.61 (6)	C9—C8—H8	120.3
N1—Cu1—Cu1 <sup>i</sup>	176.65 (6)	N1—C9—N2	115.0 (2)
C1—O1—Cu1	124.57 (18)	N1—C9—C8	121.4 (2)
C1—O2—Cu1 <sup>i</sup>	123.80 (18)	N2—C9—C8	123.6 (2)
C3—O3—Cu1	124.0 (2)	C11—C10—C15	119.4 (3)
C3—O4—Cu1 <sup>i</sup>	122.82 (18)	C11—C10—N2	117.7 (2)
C9—N1—C5	118.0 (2)	C15—C10—N2	122.7 (3)
C9—N1—Cu1	127.85 (17)	C10—C11—C12	121.6 (3)
C5—N1—Cu1	114.18 (17)	C10—C11—H11	119.2

C9—N2—C10	127.9 (2)	C12—C11—H11	119.2
C9—N2—H2	116.1	C13—C12—C11	118.2 (3)
C10—N2—H2	116.1	C13—C12—C16	121.2 (3)
O1—C1—O2	124.3 (2)	C11—C12—C16	120.6 (3)
O1—C1—C2	117.9 (2)	C14—C13—C12	120.5 (3)
O2—C1—C2	117.8 (3)	C14—C13—H13	119.8
C1—C2—H2A	109.5	C12—C13—H13	119.8
C1—C2—H2B	109.5	C13—C14—C15	121.2 (3)
H2A—C2—H2B	109.5	C13—C14—H14	119.4
C1—C2—H2C	109.5	C15—C14—H14	119.4
H2A—C2—H2C	109.5	C14—C15—C10	119.1 (3)
H2B—C2—H2C	109.5	C14—C15—H15	120.5
O4—C3—O3	125.3 (3)	C10—C15—H15	120.5
O4—C3—C4	118.0 (3)	C12—C16—H16A	109.5
O3—C3—C4	116.7 (3)	C12—C16—H16B	109.5
C3—C4—H4A	109.5	H16A—C16—H16B	109.5
C3—C4—H4B	109.5	C12—C16—H16C	109.5
H4A—C4—H4B	109.5	H16A—C16—H16C	109.5
C3—C4—H4C	109.5	H16B—C16—H16C	109.5
O4 <sup>i</sup> —Cu1—O1—C1	-84.3 (2)	C9—N1—C5—C6	-1.4 (4)
O3—Cu1—O1—C1	83.4 (2)	Cu1—N1—C5—C6	178.8 (2)
O2 <sup>i</sup> —Cu1—O1—C1	-3.6 (6)	N1—C5—C6—C7	1.3 (5)
N1—Cu1—O1—C1	177.4 (2)	C5—C6—C7—C8	-0.5 (5)
Cu1 <sup>i</sup> —Cu1—O1—C1	0.3 (2)	C6—C7—C8—C9	-0.1 (5)
O4 <sup>i</sup> —Cu1—O3—C3	0.2 (6)	C5—N1—C9—N2	-177.5 (3)
O1—Cu1—O3—C3	-85.6 (2)	Cu1—N1—C9—N2	2.2 (4)
O2 <sup>i</sup> —Cu1—O3—C3	81.7 (2)	C5—N1—C9—C8	0.7 (4)
N1—Cu1—O3—C3	179.8 (2)	Cu1—N1—C9—C8	-179.6 (2)
Cu1 <sup>i</sup> —Cu1—O3—C3	-2.0 (2)	C10—N2—C9—N1	175.0 (3)
O4 <sup>i</sup> —Cu1—N1—C9	58.8 (2)	C10—N2—C9—C8	-3.2 (5)
O3—Cu1—N1—C9	-121.1 (2)	C7—C8—C9—N1	0.0 (4)
O1—Cu1—N1—C9	148.2 (2)	C7—C8—C9—N2	178.1 (3)
O2 <sup>i</sup> —Cu1—N1—C9	-31.6 (2)	C9—N2—C10—C11	-134.6 (3)
O4 <sup>i</sup> —Cu1—N1—C5	-121.5 (2)	C9—N2—C10—C15	50.2 (5)
O3—Cu1—N1—C5	58.6 (2)	C15—C10—C11—C12	-0.2 (4)
O1—Cu1—N1—C5	-32.0 (2)	N2—C10—C11—C12	-175.6 (3)
O2 <sup>i</sup> —Cu1—N1—C5	148.2 (2)	C10—C11—C12—C13	-2.0 (4)
Cu1—O1—C1—O2	-2.2 (4)	C10—C11—C12—C16	175.8 (3)
Cu1—O1—C1—C2	178.1 (2)	C11—C12—C13—C14	2.4 (5)
Cu1 <sup>i</sup> —O2—C1—O1	3.4 (4)	C16—C12—C13—C14	-175.4 (3)
Cu1 <sup>i</sup> —O2—C1—C2	-176.9 (2)	C12—C13—C14—C15	-0.6 (5)
Cu1 <sup>i</sup> —O4—C3—O3	-5.7 (4)	C13—C14—C15—C10	-1.6 (5)
Cu1 <sup>i</sup> —O4—C3—C4	174.4 (2)	C11—C10—C15—C14	2.0 (4)
Cu1—O3—C3—O4	5.2 (4)	N2—C10—C15—C14	177.2 (3)
Cu1—O3—C3—C4	-175.0 (2)		

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

## supplementary materials

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### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N2-H2\cdots O2^i$	0.86	2.17	2.913 (3)	145.

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

Fig. 1

