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Tetra-μ-acetato-κ^8O:O'-bis{[*N*-(4-chlorophenyl)-4-methylpyridin-2-amine-κN^1]copper(II)}

Zainal A. Fairuz, Zaharah Aiyub, Zanariah Abdullah,‡ Seik Weng Ng and Edward R. T. Tiekink*

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: edward.tiekink@gmail.com

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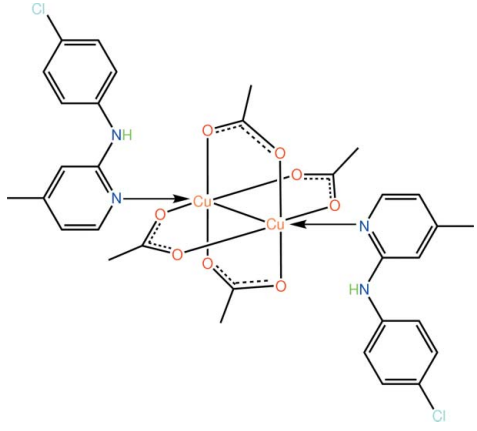
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.007 Å; R factor = 0.052; wR factor = 0.151; data-to-parameter ratio = 17.6.

In the crystal structure of the title complex, $[Cu_2(CH_3COO)_4(C_{12}H_{11}ClN_2)_2]$, the complete binuclear molecule is generated by a crystallographic centre of inversion; the four acetate groups each bridge a pair of Cu^{II} atoms. The coordination of the metal atom is distorted octahedral within a donor set defined by four O atoms, the heterocyclic N atom and the second Cu atom. The pyridine ring is twisted with respect to the benzene ring, forming a dihedral angle of 33.9 (2)°. An intramolecular N—H···O hydrogen bond is present between the amino group and a carboxyl O atom. Intermolecular interactions of the C—H···π type link molecules in the crystal structure.

Related literature

For examples of tetrakisacetatobis[(substituted 2-aminopyridyl)copper] complexes, see: Barquín *et al.* (2004); Seco *et al.* (2004); Sieroń (2004); Fairuz *et al.* (2009).



Experimental

Crystal data

$[Cu_2(C_2H_3O_2)_4(C_{12}H_{11}ClN_2)_2]$
M_r = 800.61
Monoclinic, $P2_1/c$
a = 11.7430 (17) Å
b = 15.619 (2) Å
c = 9.9866 (14) Å
β = 109.901 (2)°
V = 1722.3 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.45 mm^{-1}
T = 296 K
0.35 × 0.25 × 0.05 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.632, T_{max} = 0.931
11432 measured reflections
3951 independent reflections
2460 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.151
S = 1.04
3951 reflections
224 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.77 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cu1—O3^{i}	1.967 (3)	Cu1—O4	1.984 (3)
Cu1—O1^{i}	1.975 (3)	Cu1—N1	2.220 (3)
Cu1—O2	1.983 (3)	Cu1—Cu1^{i}	2.6431 (10)

Symmetry code: (i) $-x+2, -y+1, -z+1$.

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

*Cg*1 is the centroid of the N1,C5–C9 ring.

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O4	0.85 (4)	2.30 (2)	3.101 (5)	156 (4)
C4—H4a···*Cg*1^{ii}	0.96	2.83	3.650 (5)	144

Symmetry code: (ii) $x, -y-\frac{1}{2}, z-\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5589).

‡ Additional correspondence author, e-mail: zana@um.edu.my.

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supporting information

Acta Cryst. (2010). E**66**, m1049–m1050 [https://doi.org/10.1107/S1600536810030187]

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S1. Comment

The binuclear title complex, (I), was studied in connection with the structural characterization of tetrakisacetatobis-[(substituted 2-aminopyridyl)copper] complexes, see: Barquín *et al.*, 2004; Seco *et al.*, 2004; Sieroń, 2004; Fairuz *et al.*, 2009). The binuclear copper(II) complex, Fig. 1, is situated about a centre of inversion and features two Cu atoms bridged by four acetate groups. The Cu–O bond distances lie in the experimentally equivalent range 1.967 (3) to 1.984 (3) Å, Table 1. The coordination environment for each Cu atom is completed by a N atom derived from the *N*-4-chloro-anilino-4-picoline ligand and the second Cu atom [Cu···Cu^{i} = 2.6431 (10) Å]. The resulting hexa-coordinated geometry is based on an octahedron. An intramolecular N1–H···O4 interaction is noted, Table 2. The *N*-heterocycle is non-planar with the dihedral angle formed between the pyridine and benzene rings being 33.9 (2) °. The major twist in the molecule occurs around the amine group as seen in the value of the C9–N2–C11–C12 torsion angle of 24.1 (8) °. The most obvious intermolecular contact operating in the crystal structure is of the type C–H···π and occurs between methyl-H and pyridine rings, Table 2. These link complex molecules that stack in columns along the *a* axis, Fig. 2.

S2. Experimental

A mixture of *N*-(4-chlorophenyl)-4-methylpyridin-2-amine (0.2408 g, 1.1 mmol) in acetonitrile (15 ml) and trimethyl orthoformate (10 ml) was heated to 328 K. Copper acetate (0.1 g, 0.5 mmol) dissolved in acetonitrile (15 ml) was added drop wise to the ligand solution. The green solution was left at room temperature and green plates of (I) were collected after a few days.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.96 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 to 1.5U_{equiv}(C). The N-bound H-atom was located in a difference Fourier map, and was refined with a distance restraint of N–H 0.86±0.01 Å; the U_{iso} value was freely refined

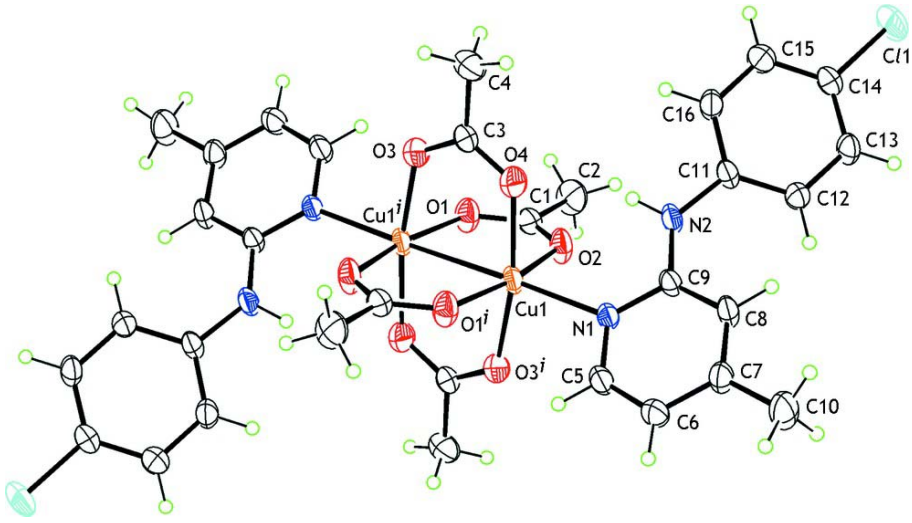

Figure 1

The molecular structure of (I) showing displacement ellipsoids at the 35% probability level. Symmetry code: (i) 2–x, 1–y, 1–z.

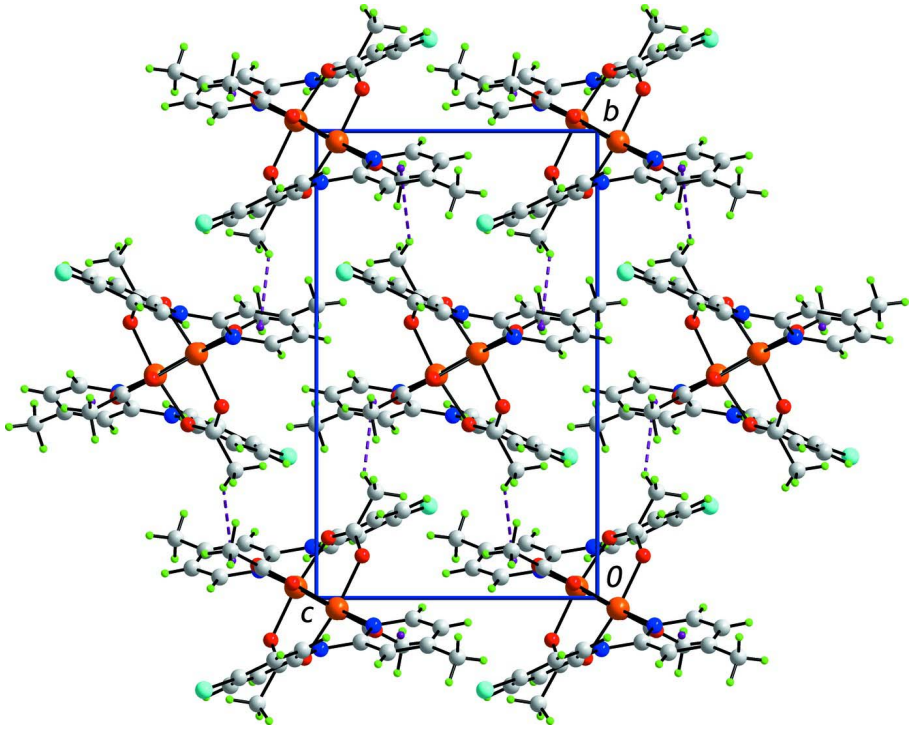

Figure 2

Unit-cell contents shown in projection down the *a* axis in (I). The C–H···π contacts are shown as purple dashed lines.

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Crystal data

$[Cu_2(C_2H_3O_2)_4(C_{12}H_{11}ClN_2)_2]$
M_r = 800.61

Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

a = 11.7430 (17) Å
b = 15.619 (2) Å
c = 9.9866 (14) Å
β = 109.901 (2)°
V = 1722.3 (4) Å^3
Z = 2
F(000) = 820
D_x = 1.544 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2098 reflections
θ = 2.5–23.4°
μ = 1.45 mm^{-1}
T = 296 K
Plate, green
0.35 × 0.25 × 0.05 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.632, T_{max} = 0.931

11432 measured reflections
3951 independent reflections
2460 reflections with $I > 2\sigma(I)$
R_{int} = 0.065
θ_{max} = 27.5°, θ_{min} = 1.8°
h = −15→13
k = −19→20
l = −11→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.151
S = 1.04
3951 reflections
224 parameters
1 restraint
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 1.063P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.77 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
Cu1	0.92999 (4)	0.47727 (4)	0.57325 (5)	0.03790 (19)
Cl1	0.17644 (11)	0.30281 (12)	0.10013 (15)	0.0756 (5)
N1	0.8173 (3)	0.4410 (2)	0.7033 (3)	0.0359 (8)
N2	0.6604 (3)	0.3905 (3)	0.5143 (4)	0.0514 (11)
H2	0.712 (3)	0.386 (3)	0.472 (4)	0.054 (14)*
O1	0.9185 (3)	0.5697 (2)	0.2909 (3)	0.0520 (8)
O2	0.7991 (2)	0.5312 (2)	0.4143 (3)	0.0526 (9)
O3	1.0247 (3)	0.4097 (2)	0.3375 (3)	0.0502 (8)
O4	0.9027 (3)	0.3711 (2)	0.4573 (4)	0.0539 (9)
C1	0.8177 (4)	0.5643 (3)	0.3087 (5)	0.0424 (10)
C2	0.7092 (4)	0.6009 (4)	0.1941 (5)	0.0656 (15)
H2A	0.6375	0.5713	0.1939	0.098*
H2B	0.7191	0.5942	0.1032	0.098*
H2C	0.7018	0.6607	0.2122	0.098*
C3	0.9545 (3)	0.3572 (3)	0.3661 (4)	0.0408 (10)
C4	0.9312 (4)	0.2732 (3)	0.2895 (5)	0.0569 (13)
H4A	0.8869	0.2365	0.3312	0.085*

H4B	1.0070	0.2468	0.2971	0.085*
H4C	0.8849	0.2825	0.1909	0.085*
C5	0.8690 (4)	0.4552 (3)	0.8442 (5)	0.0490 (12)
H5	0.9457	0.4798	0.8764	0.059*
C6	0.8160 (4)	0.4360 (4)	0.9428 (5)	0.0531 (13)
H6	0.8555	0.4474	1.0388	0.064*
C7	0.7010 (4)	0.3986 (3)	0.8961 (4)	0.0456 (11)
C8	0.6464 (3)	0.3833 (3)	0.7531 (4)	0.0417 (11)
H8	0.5700	0.3584	0.7193	0.050*
C9	0.7059 (3)	0.4054 (3)	0.6584 (4)	0.0350 (9)
C10	0.6402 (5)	0.3728 (4)	1.0011 (5)	0.0710 (17)
H10A	0.5833	0.4162	1.0044	0.107*
H10B	0.7002	0.3662	1.0939	0.107*
H10C	0.5984	0.3195	0.9718	0.107*
C11	0.5429 (3)	0.3694 (3)	0.4227 (4)	0.0375 (10)
C12	0.4356 (4)	0.3881 (3)	0.4500 (4)	0.0414 (10)
H12	0.4400	0.4149	0.5347	0.050*
C13	0.3230 (4)	0.3668 (3)	0.3517 (5)	0.0447 (11)
H13	0.2526	0.3776	0.3717	0.054*
C14	0.3172 (4)	0.3294 (3)	0.2243 (5)	0.0436 (11)
C15	0.4220 (4)	0.3123 (3)	0.1936 (5)	0.0500 (12)
H15	0.4171	0.2877	0.1070	0.060*
C16	0.5334 (4)	0.3321 (3)	0.2928 (5)	0.0474 (12)
H16	0.6034	0.3202	0.2725	0.057*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0227 (2)	0.0514 (4)	0.0388 (3)	−0.0041 (2)	0.00946 (19)	0.0002 (3)
Cl1	0.0348 (6)	0.1143 (13)	0.0618 (8)	−0.0131 (7)	−0.0044 (5)	−0.0148 (8)
N1	0.0246 (15)	0.050 (2)	0.0316 (17)	−0.0048 (15)	0.0081 (13)	−0.0007 (16)
N2	0.0275 (17)	0.091 (3)	0.037 (2)	−0.021 (2)	0.0132 (15)	−0.011 (2)
O1	0.0338 (15)	0.075 (2)	0.0466 (18)	0.0082 (16)	0.0124 (14)	0.0134 (17)
O2	0.0260 (14)	0.080 (3)	0.0495 (18)	0.0036 (15)	0.0100 (13)	0.0128 (17)
O3	0.0449 (17)	0.058 (2)	0.0533 (19)	−0.0118 (16)	0.0234 (15)	−0.0082 (16)
O4	0.0485 (18)	0.058 (2)	0.064 (2)	−0.0131 (16)	0.0299 (17)	−0.0089 (17)
C1	0.031 (2)	0.049 (3)	0.043 (2)	0.001 (2)	0.0071 (18)	−0.005 (2)
C2	0.043 (3)	0.085 (4)	0.056 (3)	0.023 (3)	0.002 (2)	0.010 (3)
C3	0.0264 (18)	0.050 (3)	0.041 (2)	−0.0003 (19)	0.0048 (17)	−0.003 (2)
C4	0.053 (3)	0.052 (3)	0.061 (3)	−0.012 (2)	0.013 (2)	−0.008 (3)
C5	0.033 (2)	0.074 (4)	0.039 (2)	−0.017 (2)	0.0113 (18)	−0.007 (2)
C6	0.042 (2)	0.081 (4)	0.036 (2)	−0.017 (3)	0.0125 (19)	−0.011 (2)
C7	0.033 (2)	0.069 (3)	0.036 (2)	−0.004 (2)	0.0133 (17)	0.005 (2)
C8	0.0242 (18)	0.059 (3)	0.041 (2)	−0.0074 (19)	0.0093 (17)	0.006 (2)
C9	0.0247 (17)	0.043 (3)	0.033 (2)	−0.0035 (17)	0.0049 (15)	0.0015 (18)
C10	0.051 (3)	0.121 (5)	0.045 (3)	−0.016 (3)	0.021 (2)	0.005 (3)
C11	0.0256 (18)	0.049 (3)	0.035 (2)	−0.0080 (18)	0.0068 (16)	0.0046 (19)
C12	0.033 (2)	0.054 (3)	0.037 (2)	−0.002 (2)	0.0110 (18)	−0.004 (2)

C13	0.0257 (19)	0.056 (3)	0.050 (3)	0.005 (2)	0.0102 (18)	0.003 (2)
C14	0.030 (2)	0.051 (3)	0.044 (2)	−0.004 (2)	0.0039 (17)	−0.001 (2)
C15	0.039 (2)	0.065 (3)	0.041 (2)	0.000 (2)	0.0069 (19)	−0.006 (2)
C16	0.030 (2)	0.073 (4)	0.040 (2)	−0.004 (2)	0.0140 (18)	−0.001 (2)

Geometric parameters (Å, º)

Cu1—O3^{i}	1.967 (3)	C4—H4B	0.9600
Cu1—O1^{i}	1.975 (3)	C4—H4C	0.9600
Cu1—O2	1.983 (3)	C5—C6	1.366 (6)
Cu1—O4	1.984 (3)	C5—H5	0.9300
Cu1—N1	2.220 (3)	C6—C7	1.397 (6)
Cu1—Cu1^{i}	2.6431 (10)	C6—H6	0.9300
Cl1—C14	1.745 (4)	C7—C8	1.373 (6)
N1—C9	1.350 (5)	C7—C10	1.511 (6)
N1—C5	1.348 (5)	C8—C9	1.397 (5)
N2—C9	1.373 (5)	C8—H8	0.9300
N2—C11	1.411 (5)	C10—H10A	0.9600
N2—H2	0.85 (4)	C10—H10B	0.9600
O1—C1	1.258 (5)	C10—H10C	0.9600
O1—Cu1^{i}	1.975 (3)	C11—C16	1.392 (6)
O2—C1	1.259 (5)	C11—C12	1.407 (6)
O3—C3	1.262 (5)	C12—C13	1.393 (5)
O3—Cu1^{i}	1.967 (3)	C12—H12	0.9300
O4—C3	1.274 (5)	C13—C14	1.380 (6)
C1—C2	1.506 (6)	C13—H13	0.9300
C2—H2A	0.9600	C14—C15	1.392 (6)
C2—H2B	0.9600	C15—C16	1.381 (6)
C2—H2C	0.9600	C15—H15	0.9300
C3—C4	1.497 (6)	C16—H16	0.9300
C4—H4A	0.9600		

O3^{i}—Cu1—O1^{i}	88.89 (14)	H4A—C4—H4C	109.5
O3^{i}—Cu1—O2	89.88 (14)	H4B—C4—H4C	109.5
O1^{i}—Cu1—O2	168.34 (12)	N1—C5—C6	124.2 (4)
O3^{i}—Cu1—O4	168.17 (12)	N1—C5—H5	117.9
O1^{i}—Cu1—O4	91.08 (15)	C6—C5—H5	117.9
O2—Cu1—O4	87.76 (14)	C5—C6—C7	118.5 (4)
O3^{i}—Cu1—N1	95.02 (13)	C5—C6—H6	120.7
O1^{i}—Cu1—N1	94.59 (12)	C7—C6—H6	120.7
O2—Cu1—N1	97.06 (12)	C8—C7—C6	118.4 (4)
O4—Cu1—N1	96.77 (13)	C8—C7—C10	120.8 (4)
O3^{i}—Cu1—Cu1^{i}	83.47 (9)	C6—C7—C10	120.8 (4)
O1^{i}—Cu1—Cu1^{i}	84.00 (9)	C7—C8—C9	119.8 (4)
O2—Cu1—Cu1^{i}	84.35 (9)	C7—C8—H8	120.1
O4—Cu1—Cu1^{i}	84.76 (9)	C9—C8—H8	120.1
N1—Cu1—Cu1^{i}	177.94 (9)	N1—C9—N2	113.9 (3)
C9—N1—C5	117.2 (3)	N1—C9—C8	121.9 (3)

C9—N1—Cu1	127.9 (3)	N2—C9—C8	124.2 (4)
C5—N1—Cu1	115.0 (3)	C7—C10—H10A	109.5
C9—N2—C11	131.6 (3)	C7—C10—H10B	109.5
C9—N2—H2	116 (3)	H10A—C10—H10B	109.5
C11—N2—H2	111 (3)	C7—C10—H10C	109.5
C1—O1—Cu1^{i}	123.3 (3)	H10A—C10—H10C	109.5
C1—O2—Cu1	122.5 (3)	H10B—C10—H10C	109.5
C3—O3—Cu1^{i}	125.2 (3)	C16—C11—N2	116.9 (4)
C3—O4—Cu1	122.5 (3)	C16—C11—C12	118.3 (4)
O1—C1—O2	125.9 (4)	N2—C11—C12	124.6 (4)
O1—C1—C2	117.4 (4)	C13—C12—C11	120.8 (4)
O2—C1—C2	116.7 (4)	C13—C12—H12	119.6
C1—C2—H2A	109.5	C11—C12—H12	119.6
C1—C2—H2B	109.5	C14—C13—C12	119.2 (4)
H2A—C2—H2B	109.5	C14—C13—H13	120.4
C1—C2—H2C	109.5	C12—C13—H13	120.4
H2A—C2—H2C	109.5	C13—C14—C15	120.9 (4)
H2B—C2—H2C	109.5	C13—C14—Cl1	119.6 (3)
O3—C3—O4	123.9 (4)	C15—C14—Cl1	119.5 (3)
O3—C3—C4	118.2 (4)	C16—C15—C14	119.5 (4)
O4—C3—C4	117.8 (4)	C16—C15—H15	120.3
C3—C4—H4A	109.5	C14—C15—H15	120.3
C3—C4—H4B	109.5	C15—C16—C11	121.2 (4)
H4A—C4—H4B	109.5	C15—C16—H16	119.4
C3—C4—H4C	109.5	C11—C16—H16	119.4

O3^{i}—Cu1—N1—C9	134.2 (4)	Cu1—N1—C5—C6	−178.7 (4)
O1^{i}—Cu1—N1—C9	−136.5 (4)	N1—C5—C6—C7	0.3 (8)
O2—Cu1—N1—C9	43.7 (4)	C5—C6—C7—C8	−0.3 (8)
O4—Cu1—N1—C9	−44.9 (4)	C5—C6—C7—C10	177.6 (5)
O3^{i}—Cu1—N1—C5	−47.1 (3)	C6—C7—C8—C9	−0.2 (7)
O1^{i}—Cu1—N1—C5	42.2 (3)	C10—C7—C8—C9	−178.1 (5)
O2—Cu1—N1—C5	−137.6 (3)	C5—N1—C9—N2	−178.3 (4)
O4—Cu1—N1—C5	133.8 (3)	Cu1—N1—C9—N2	0.4 (6)
O3^{i}—Cu1—O2—C1	84.2 (4)	C5—N1—C9—C8	−0.6 (6)
O1^{i}—Cu1—O2—C1	0.3 (9)	Cu1—N1—C9—C8	178.0 (3)
O4—Cu1—O2—C1	−84.2 (4)	C11—N2—C9—N1	−167.2 (5)
N1—Cu1—O2—C1	179.2 (4)	C11—N2—C9—C8	15.3 (8)
Cu1^{i}—Cu1—O2—C1	0.8 (4)	C7—C8—C9—N1	0.7 (7)
O3^{i}—Cu1—O4—C3	7.9 (9)	C7—C8—C9—N2	178.1 (5)
O1^{i}—Cu1—O4—C3	−81.8 (3)	C9—N2—C11—C16	−159.9 (5)
O2—Cu1—O4—C3	86.6 (3)	C9—N2—C11—C12	24.1 (8)
N1—Cu1—O4—C3	−176.6 (3)	C16—C11—C12—C13	2.3 (7)
Cu1^{i}—Cu1—O4—C3	2.0 (3)	N2—C11—C12—C13	178.3 (4)
Cu1^{i}—O1—C1—O2	2.0 (7)	C11—C12—C13—C14	−2.1 (7)
Cu1^{i}—O1—C1—C2	−178.3 (3)	C12—C13—C14—C15	0.5 (7)
Cu1—O2—C1—O1	−1.8 (7)	C12—C13—C14—Cl1	−179.8 (4)
Cu1—O2—C1—C2	178.5 (3)	C13—C14—C15—C16	0.8 (8)

Cu1^{i}—O3—C3—O4	0.4 (6)	Cl1—C14—C15—C16	−178.9 (4)
Cu1^{i}—O3—C3—C4	−179.1 (3)	C14—C15—C16—C11	−0.5 (8)
Cu1—O4—C3—O3	−2.1 (6)	N2—C11—C16—C15	−177.3 (4)
Cu1—O4—C3—C4	177.4 (3)	C12—C11—C16—C15	−1.0 (7)
C9—N1—C5—C6	0.1 (7)		

Symmetry code: (i) −*x*+2, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the N1,C5–C9 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O4	0.85 (4)	2.30 (2)	3.101 (5)	156 (4)
C4—H4a···*Cg*1^{ii}	0.96	2.83	3.650 (5)	144

Symmetry code: (ii) *x*, −*y*−1/2, *z*−3/2.