

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

[5-Chloro-2-hydroxy-*N'*-(2-oxidobenzylidene)benzohydrazidato]pyridine-copper(II)

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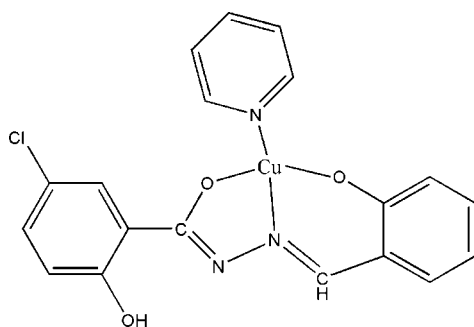
Received 27 May 2009; accepted 29 May 2009

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.011$ Å; R factor = 0.040; wR factor = 0.116; data-to-parameter ratio = 9.3.

In the title complex, $[\text{Cu}(\text{C}_{14}\text{H}_9\text{ClN}_2\text{O}_3)(\text{C}_5\text{H}_5\text{N})]$, the Cu^{II} ion exhibits a distorted *trans*- CuN_2O_2 square-planar geometry arising from the *O,O,N*-tridentate ligand and a pyridine molecule. An intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond occurs. In the crystal structure, weak intermolecular $\text{C}-\text{H}\cdots\pi$ interactions generate a chain. The crystal studied was an inversion twin.

Related literature

For background on the coordination chemistry of salicylaldehyde-type ligands, see: Bai *et al.* (2005). For information on $\text{C}-\text{H}\cdots\pi$ interactions, see: Nishio (2004).



Experimental

Crystal data

$[\text{Cu}(\text{C}_{14}\text{H}_9\text{ClN}_2\text{O}_3)(\text{C}_5\text{H}_5\text{N})]$
 $M_r = 431.32$
 Monoclinic, *Cc*

$a = 23.586$ (2) Å
 $b = 4.8268$ (6) Å
 $c = 17.88540$ (18) Å

$\beta = 120.809$ (2)°
 $V = 1748.8$ (3) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 1.43$ mm⁻¹
 $T = 298$ K
 $0.39 \times 0.28 \times 0.17$ mm

Data collection

Siemens SMART CCD diffractometer
 Absorption correction: multi-scan (*SADABS*; Siemens, 1996)
 $T_{\text{min}} = 0.606$, $T_{\text{max}} = 0.793$

4087 measured reflections
 2273 independent reflections
 1849 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.116$
 $S = 1.00$
 2273 reflections
 244 parameters
 2 restraints

H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.37$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³
 Absolute structure: Flack (1983),
 725 Friedel pairs
 Flack parameter: 0.50 (2)

Table 1

Selected bond lengths (Å).

Cu1—O3	1.897 (4)	Cu1—N2	1.945 (6)
Cu1—O1	1.934 (4)	Cu1—N3	1.965 (6)

Table 2

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O2}-\text{H2}\cdots\text{N1}$	0.82	1.85	2.575 (9)	147
$\text{C16}-\text{H16}\cdots\text{Cg1}^i$	0.93	2.81	3.48 (3)	130

Symmetry code: (i) $x, -y + 1, z - \frac{1}{2}$. Cg1 is the centroid of the C9–C14 ring.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; 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: *SHELXTL*.

We acknowledge the National Natural Science Foundation of China (grant No. 20771053) and the Natural Science Foundation of Shandong Province (grant No. 2005ZX09) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2989).

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supplementary materials

Acta Cryst. (2009). E65, m738 [doi:10.1107/S1600536809020546]

[5-Chloro-2-hydroxy-*N'*-(2-oxidobenzylidene)benzohydrazidato]pyridinecopper(II)

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Comment

The chemistry of aroylhydrazones has gained a special attraction due to their coordination abilities to metal ions (Bai *et al.*, 2005). However, researches on the complexes with salicylaldehyde-5-chlorosalicylichydrazone have not reported. So we have synthesized a new complex (Fig. 1), which has been characterized by X-ray diffraction and elemental analysis. The structure of the title complex, (I), contains one ligand molecule, one pyridine molecule and one copper(II). The copper(II) coordination environment in the complex exhibits a distorted quadrilateral geometry (Table 1). In the crystal packing, the complex molecules are linked into one-dimensional chain by intermolecular C—H \cdots π interactions (Nishio, 2004) (Table 2, Fig. 2).

Experimental

A solution of salicylaldehyde (1.46 g, 12 mmol) in ethanol (10 ml) was added to a solution of 5-chlorosalicylichydrazine (1.87 g, 10 mmol) in ethanol (10 ml). The mixture was refluxed for 3 h, and then the precipitate was collected, washed several times with ethanol and dried *in vacuo* (yield 75.6%). m.p. > 300 K. A solution of Cu(OAc)₂ (0.04 g, 0.2 mmol) in methanol (10 ml) was added to the mixture of salicylaldehyde-5-chlorosalicylichydrazone (0.058 g, 0.2 mmol) and sodium methylate (0.0324 g, 0.6 mmol) in pyridine (10 ml). A green solution was obtained after stirring for 4 h. After being filtrated, dimethyl ether was slowly diffused into the filtrate, then green blocks of (I) were obtained after several weeks (m.p. > 400 K) Elemental analysis calculated for C₁₉H₁₄ClN₃O₃Cu₁: C, 52.90; H, 3.27; N, 9.74. Found (%): C, 52.95; H, 3.19; N, 9.69

Refinement

The H atoms were positioned with idealized geometry (C—H = 0.93 Å, O—H = 0.82 Å) and were refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$.

Figures

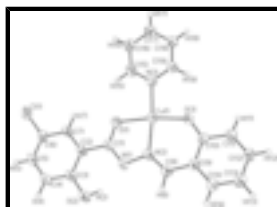


Fig. 1. The molecular structure of (I), showing 40% probability displacement ellipsoids. H atoms have been omitted for clarity.

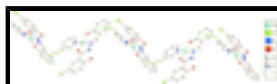


Fig. 2. View of the chains in (I). Intermolecular C—H \cdots π are shown as dashed lines. Most of H atoms are omitted.

[5-Chloro-2-hydroxy-*N*'-(2-oxidobenzylidene)benzohydrazidato]pyridinecopper(II)

Crystal data

[Cu(C ₁₄ H ₉ ClN ₂ O ₃)(C ₅ H ₅ N)]	$F_{000} = 876$
$M_r = 431.32$	$D_x = 1.638 \text{ Mg m}^{-3}$
Monoclinic, <i>Cc</i>	Mo $K\alpha$ radiation
$a = 23.586 (2) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 4.8268 (6) \text{ \AA}$	Cell parameters from 1781 reflections
$c = 17.88540 (18) \text{ \AA}$	$\theta = 2.7\text{--}23.7^\circ$
$\beta = 120.809 (2)^\circ$	$\mu = 1.43 \text{ mm}^{-1}$
$V = 1748.8 (3) \text{ \AA}^3$	$T = 298 \text{ K}$
$Z = 4$	Block, green
	$0.39 \times 0.28 \times 0.17 \text{ mm}$

Data collection

Siemens SMART CCD diffractometer	2273 independent reflections
Radiation source: fine-focus sealed tube	1849 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.021$
$T = 298 \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
ω scans	$\theta_{\text{min}} = 2.0^\circ$
Absorption correction: multi-scan (SADABS; Siemens, 1996)	$h = -28 \rightarrow 25$
$T_{\text{min}} = 0.606$, $T_{\text{max}} = 0.793$	$k = -5 \rightarrow 5$
4087 measured reflections	$l = -17 \rightarrow 21$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.0729P)^2 + 0.8658P]$
$wR(F^2) = 0.116$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{\text{max}} < 0.001$
2273 reflections	$\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$
244 parameters	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 725 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.50 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu1	0.18179 (4)	0.48947 (15)	0.25227 (4)	0.0455 (2)
Cl1	0.00840 (11)	1.5179 (4)	-0.07019 (13)	0.0697 (5)
N1	0.0517 (3)	0.6161 (12)	0.2113 (3)	0.0462 (13)
N2	0.1052 (3)	0.4396 (12)	0.2649 (4)	0.0452 (14)
N3	0.2566 (3)	0.5850 (12)	0.2378 (4)	0.0487 (13)
O1	0.1235 (2)	0.7588 (9)	0.1676 (3)	0.0519 (11)
O2	-0.0618 (2)	0.8534 (11)	0.1447 (4)	0.0721 (14)
H2	-0.0319	0.7470	0.1764	0.108*
O3	0.2275 (2)	0.1945 (8)	0.3304 (3)	0.0509 (11)
C1	0.0675 (3)	0.7694 (13)	0.1643 (4)	0.0468 (15)
C2	0.0182 (3)	0.9709 (11)	0.1036 (4)	0.0438 (14)
C3	-0.0435 (4)	1.0041 (13)	0.0969 (5)	0.0545 (17)
C4	-0.0874 (3)	1.1994 (15)	0.0388 (5)	0.064 (2)
H4	-0.1282	1.2236	0.0343	0.076*
C5	-0.0718 (3)	1.3556 (16)	-0.0116 (5)	0.0611 (19)
H5	-0.1020	1.4840	-0.0503	0.073*
C6	-0.0118 (3)	1.3239 (12)	-0.0053 (4)	0.0502 (15)
C7	0.0333 (3)	1.1330 (13)	0.0522 (4)	0.0478 (15)
H7	0.0740	1.1134	0.0564	0.057*
C8	0.0986 (3)	0.2719 (14)	0.3155 (4)	0.0494 (16)
H8	0.0590	0.2752	0.3150	0.059*
C9	0.1478 (3)	0.0818 (12)	0.3722 (4)	0.0450 (15)
C10	0.2086 (3)	0.0513 (12)	0.3749 (4)	0.0460 (15)
C11	0.2519 (3)	-0.1564 (13)	0.4325 (5)	0.0539 (16)
H11	0.2920	-0.1860	0.4357	0.065*
C12	0.2365 (3)	-0.3133 (13)	0.4832 (4)	0.0576 (17)
H12	0.2661	-0.4472	0.5197	0.069*
C13	0.1771 (4)	-0.2753 (13)	0.4808 (4)	0.0579 (19)
H13	0.1671	-0.3797	0.5162	0.069*
C14	0.1341 (4)	-0.0825 (14)	0.4255 (5)	0.0540 (16)
H14	0.0940	-0.0587	0.4230	0.065*
C15	0.2503 (4)	0.7717 (17)	0.1806 (5)	0.069 (2)
H15	0.2091	0.8520	0.1452	0.083*

supplementary materials

C16	0.3020 (4)	0.8541 (18)	0.1705 (6)	0.076 (2)
H16	0.2958	0.9905	0.1302	0.092*
C17	0.3626 (3)	0.7324 (16)	0.2205 (5)	0.0635 (18)
H17	0.3981	0.7803	0.2143	0.076*
C18	0.3687 (4)	0.5405 (17)	0.2790 (6)	0.080 (3)
H18	0.4090	0.4535	0.3143	0.095*
C19	0.3141 (4)	0.4733 (15)	0.2863 (6)	0.068 (2)
H19	0.3193	0.3431	0.3277	0.082*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0378 (3)	0.0493 (4)	0.0461 (4)	0.0027 (3)	0.0192 (3)	-0.0002 (4)
Cl1	0.0771 (12)	0.0676 (12)	0.0674 (12)	0.0189 (9)	0.0392 (10)	0.0148 (9)
N1	0.039 (3)	0.044 (3)	0.046 (3)	0.012 (3)	0.015 (3)	0.000 (3)
N2	0.036 (3)	0.052 (3)	0.038 (3)	0.000 (3)	0.012 (3)	-0.010 (3)
N3	0.045 (3)	0.051 (3)	0.053 (4)	0.002 (3)	0.027 (3)	0.002 (3)
O1	0.041 (2)	0.061 (3)	0.053 (3)	0.007 (2)	0.024 (2)	0.008 (2)
O2	0.060 (3)	0.079 (3)	0.090 (4)	0.019 (3)	0.048 (3)	0.013 (3)
O3	0.045 (2)	0.046 (2)	0.060 (3)	0.007 (2)	0.026 (2)	0.005 (2)
C1	0.041 (3)	0.045 (3)	0.044 (4)	0.002 (3)	0.013 (3)	-0.010 (3)
C2	0.040 (3)	0.044 (3)	0.044 (3)	0.004 (3)	0.019 (3)	-0.011 (3)
C3	0.047 (4)	0.057 (4)	0.061 (4)	0.005 (3)	0.028 (3)	-0.004 (3)
C4	0.046 (3)	0.068 (5)	0.072 (5)	0.013 (3)	0.027 (4)	-0.006 (4)
C5	0.049 (4)	0.062 (4)	0.057 (5)	0.020 (4)	0.016 (3)	-0.003 (4)
C6	0.054 (4)	0.045 (4)	0.043 (4)	0.006 (3)	0.019 (3)	-0.006 (3)
C7	0.041 (3)	0.049 (4)	0.049 (4)	0.005 (3)	0.019 (3)	-0.012 (3)
C8	0.045 (3)	0.054 (4)	0.054 (4)	-0.004 (3)	0.030 (3)	-0.009 (3)
C9	0.053 (3)	0.035 (3)	0.048 (4)	-0.003 (3)	0.026 (3)	-0.010 (3)
C10	0.048 (3)	0.038 (3)	0.048 (4)	-0.006 (3)	0.021 (3)	-0.010 (3)
C11	0.054 (4)	0.040 (3)	0.065 (4)	-0.005 (3)	0.028 (3)	-0.006 (3)
C12	0.060 (4)	0.043 (4)	0.057 (4)	0.003 (3)	0.021 (3)	-0.001 (3)
C13	0.069 (4)	0.053 (4)	0.050 (5)	-0.013 (4)	0.028 (4)	-0.005 (3)
C14	0.060 (4)	0.051 (4)	0.053 (4)	-0.003 (3)	0.031 (3)	-0.003 (3)
C15	0.050 (4)	0.083 (5)	0.067 (5)	0.015 (4)	0.024 (4)	0.017 (4)
C16	0.078 (5)	0.083 (5)	0.084 (6)	0.018 (5)	0.053 (5)	0.031 (5)
C17	0.052 (4)	0.081 (5)	0.062 (4)	-0.001 (4)	0.031 (3)	0.000 (4)
C18	0.047 (4)	0.095 (6)	0.092 (6)	0.009 (4)	0.033 (4)	0.033 (5)
C19	0.049 (4)	0.074 (6)	0.072 (6)	0.002 (3)	0.024 (4)	0.023 (4)

Geometric parameters (\AA , $^\circ$)

Cu1—O3	1.897 (4)	C7—H7	0.9300
Cu1—O1	1.934 (4)	C8—C9	1.419 (9)
Cu1—N2	1.945 (6)	C8—H8	0.9300
Cu1—N3	1.965 (6)	C9—C14	1.401 (10)
Cl1—C6	1.737 (7)	C9—C10	1.416 (9)
N1—C1	1.310 (9)	C10—C11	1.426 (10)
N1—N2	1.413 (8)	C11—C12	1.364 (10)

N2—C8	1.282 (9)	C11—H11	0.9300
N3—C19	1.296 (10)	C12—C13	1.393 (10)
N3—C15	1.313 (9)	C12—H12	0.9300
O1—C1	1.292 (7)	C13—C14	1.359 (9)
O2—C3	1.351 (9)	C13—H13	0.9300
O2—H2	0.8200	C14—H14	0.9300
O3—C10	1.293 (8)	C15—C16	1.380 (11)
C1—C2	1.478 (8)	C15—H15	0.9300
C2—C7	1.386 (10)	C16—C17	1.370 (10)
C2—C3	1.405 (10)	C16—H16	0.9300
C3—C4	1.393 (10)	C17—C18	1.349 (11)
C4—C5	1.362 (11)	C17—H17	0.9300
C4—H4	0.9300	C18—C19	1.396 (12)
C5—C6	1.370 (10)	C18—H18	0.9300
C5—H5	0.9300	C19—H19	0.9300
C6—C7	1.385 (9)		
O3—Cu1—O1	171.5 (2)	N2—C8—C9	124.1 (6)
O3—Cu1—N2	91.9 (2)	N2—C8—H8	117.9
O1—Cu1—N2	81.0 (2)	C9—C8—H8	117.9
O3—Cu1—N3	93.6 (2)	C14—C9—C10	120.1 (6)
O1—Cu1—N3	93.7 (2)	C14—C9—C8	117.5 (6)
N2—Cu1—N3	173.5 (3)	C10—C9—C8	122.4 (6)
C1—N1—N2	109.1 (5)	O3—C10—C9	125.8 (6)
C8—N2—N1	118.0 (6)	O3—C10—C11	118.4 (6)
C8—N2—Cu1	128.0 (5)	C9—C10—C11	115.8 (6)
N1—N2—Cu1	114.0 (4)	C12—C11—C10	122.4 (7)
C19—N3—C15	118.0 (7)	C12—C11—H11	118.8
C19—N3—Cu1	121.1 (6)	C10—C11—H11	118.8
C15—N3—Cu1	120.8 (5)	C11—C12—C13	120.8 (6)
C1—O1—Cu1	111.3 (4)	C11—C12—H12	119.6
C3—O2—H2	109.5	C13—C12—H12	119.6
C10—O3—Cu1	127.5 (4)	C14—C13—C12	118.5 (6)
O1—C1—N1	124.6 (5)	C14—C13—H13	120.7
O1—C1—C2	117.5 (6)	C12—C13—H13	120.7
N1—C1—C2	117.9 (5)	C13—C14—C9	122.4 (7)
C7—C2—C3	119.1 (6)	C13—C14—H14	118.8
C7—C2—C1	119.0 (6)	C9—C14—H14	118.8
C3—C2—C1	121.9 (6)	N3—C15—C16	123.0 (7)
O2—C3—C4	118.6 (7)	N3—C15—H15	118.5
O2—C3—C2	122.5 (6)	C16—C15—H15	118.5
C4—C3—C2	118.9 (7)	C17—C16—C15	119.1 (7)
C5—C4—C3	121.2 (7)	C17—C16—H16	120.4
C5—C4—H4	119.4	C15—C16—H16	120.4
C3—C4—H4	119.4	C18—C17—C16	117.6 (7)
C4—C5—C6	120.1 (6)	C18—C17—H17	121.2
C4—C5—H5	120.0	C16—C17—H17	121.2
C6—C5—H5	120.0	C17—C18—C19	119.6 (7)
C5—C6—C7	120.3 (7)	C17—C18—H18	120.2
C5—C6—C11	120.7 (5)	C19—C18—H18	120.2

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C7—C6—C11	119.0 (5)	N3—C19—C18	122.7 (8)
C6—C7—C2	120.5 (6)	N3—C19—H19	118.7
C6—C7—H7	119.8	C18—C19—H19	118.7
C2—C7—H7	119.8		
C1—N1—N2—C8	-179.6 (6)	O1—C1—C2—C3	-178.1 (6)
C1—N1—N2—Cu1	1.2 (6)	N1—C1—C2—C3	1.3 (9)
O3—Cu1—N2—C8	4.3 (6)	C7—C2—C3—O2	179.9 (6)
O1—Cu1—N2—C8	179.6 (6)	C7—C2—C3—C4	-0.1 (9)
N3—Cu1—N2—C8	-143 (2)	C1—C2—C3—C4	179.5 (6)
O3—Cu1—N2—N1	-176.6 (4)	O2—C3—C4—C5	-179.4 (7)
O1—Cu1—N2—N1	-1.2 (4)	C4—C5—C6—C11	179.1 (6)
N3—Cu1—N2—N1	36 (3)	C5—C6—C7—C2	0.4 (9)
O3—Cu1—N3—C19	-9.1 (7)	C11—C6—C7—C2	-178.7 (5)
O1—Cu1—N3—C19	175.1 (7)	C3—C2—C7—C6	-0.4 (9)
N2—Cu1—N3—C19	139 (2)	C1—C2—C7—C6	-180.0 (5)
O3—Cu1—N3—C15	173.4 (6)	N1—N2—C8—C9	179.5 (5)
O1—Cu1—N3—C15	-2.5 (6)	N2—C8—C9—C14	178.1 (6)
N2—Cu1—N3—C15	-39 (3)	Cu1—O3—C10—C9	1.9 (9)
O3—Cu1—O1—C1	34.5 (18)	Cu1—O3—C10—C11	-177.8 (4)
N2—Cu1—O1—C1	1.0 (4)	C14—C9—C10—O3	-178.4 (6)
N3—Cu1—O1—C1	-175.1 (4)	C8—C9—C10—O3	2.9 (9)
O1—Cu1—O3—C10	-37.5 (19)	C14—C9—C10—C11	1.3 (8)
N2—Cu1—O3—C10	-4.5 (5)	C8—C9—C10—C11	-177.4 (6)
N3—Cu1—O3—C10	172.0 (5)	O3—C10—C11—C12	178.6 (6)
Cu1—O1—C1—N1	-0.6 (7)	C9—C10—C11—C12	-1.0 (9)
Cu1—O1—C1—C2	178.7 (4)	C8—C9—C14—C13	178.5 (6)
N2—N1—C1—O1	-0.4 (8)	Cu1—N3—C15—C16	177.2 (7)
N2—N1—C1—C2	-179.7 (5)	N3—C15—C16—C17	1.7 (14)
O1—C1—C2—C7	1.4 (8)	Cu1—N3—C19—C18	-178.7 (7)
N1—C1—C2—C7	-179.2 (6)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2 \cdots N1	0.82	1.85	2.575 (9)	147
C16—H16 \cdots Cg1 ⁱ	0.93	2.81	3.48 (3)	130

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

Fig. 1

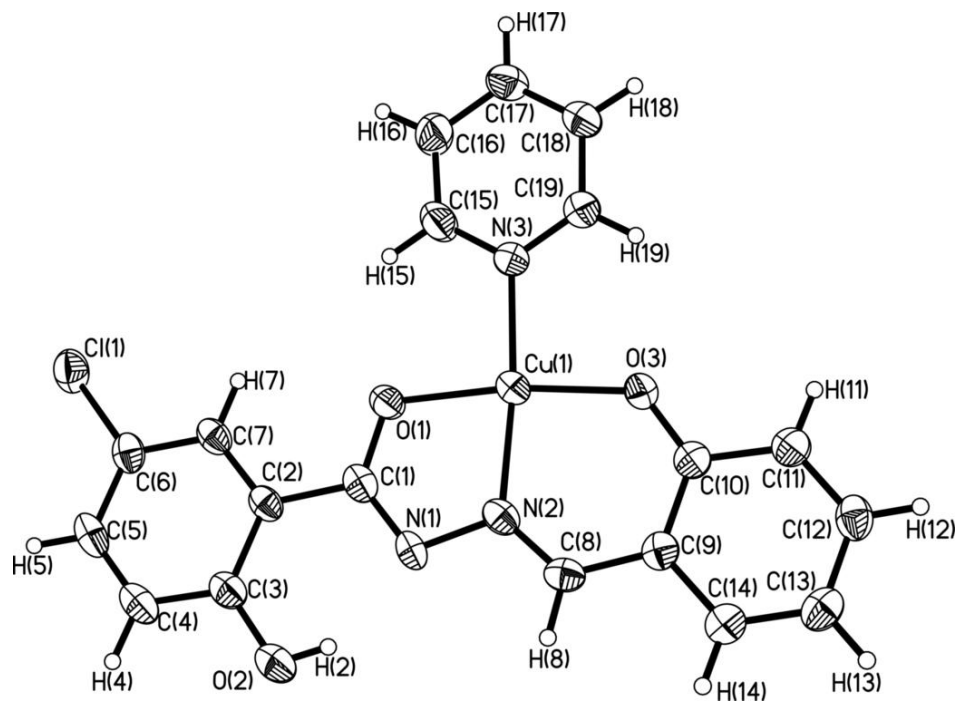


Fig. 2

