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Aqua(2,2′-bipyridine-κ^2N,N')(pyridine-2,6-dicarboxylate *N*-oxide-κ^2O^1,O^2) copper(II) trihydrate

Xin-Yan Zhang

Li Shui Vocational & Technical College, Lishui, Zhejiang 323000, People's Republic of China
Correspondence e-mail: zjlsxa@126.com

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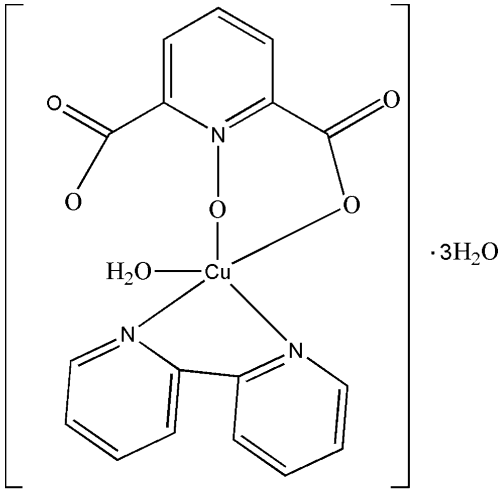
Key indicators: single-crystal X-ray study; T = 297 K; mean σ(C–C) = 0.008 Å; R factor = 0.045; wR factor = 0.140; data-to-parameter ratio = 14.6.

In the title complex, $[Cu(C_7H_3NO_5)(C_{10}H_8N_2)(H_2O)]\cdot 3H_2O$, the Cu^{II} atom has a slightly distorted square-pyramidal coordination geometry, with a basal plane formed by two N atoms of the 2,2′-bipyridine ligand and two O atoms of the pyridine-2,6-dicarboxylate *N*-oxide. The apical position is occupied by a water molecule. The crystal structure contains O—H···O intermolecular hydrogen bonds.

Related literature

For related literature, see: Moulton & Zaworotko (2001); Paul (1984); Wen *et al.* (2005).



Experimental

Crystal data

$[Cu(C_7H_3NO_5)(C_{10}H_8N_2)(H_2O)]\cdot 3H_2O$
M_r = 472.89
Triclinic, $P\overline{1}$
a = 6.8293 (2) Å
b = 11.8827 (4) Å
c = 12.5673 (4) Å
α = 86.136 (2)°
β = 81.584 (2)°
γ = 86.151 (2)°
V = 1004.87 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.14 mm^{-1}
T = 297 (2) K
0.20 × 0.18 × 0.10 mm

Data collection

Bruker SMART APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.804, T_{max} = 0.894
13440 measured reflections
3953 independent reflections
3133 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.140
S = 1.03
3953 reflections
271 parameters
12 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O7^{i}	0.85	1.94	2.733 (5)	154
O1—H1B···O5^{i}	0.85	2.09	2.867 (5)	152
O1—H1B···O4^{i}	0.85	2.53	3.265 (5)	144
O7—H7A···O3^{ii}	0.86	1.93	2.780 (6)	171
O7—H7B···O2^{i}	0.86	1.97	2.813 (5)	166
O8—H8A···O2^{i}	0.89	1.88	2.716 (8)	157
O8—H8B···O9	0.97	2.17	2.735 (12)	116
O9—H9A···O3^{ii}	0.89	1.92	2.768 (7)	158
O9—H9B···O9^{iii}	0.89	2.46	2.930 (19)	113

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HY2059).

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supplementary materials

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Aqua(2,2'-bipyridine-κ^2N,N')(pyridine-2,6-dicarboxylate *N*-oxide-κ^2O^1,O^2)copper(II) trihydrate

X.-Y. Zhang

Comment

Many of the efforts have so far devoted to the study of transition-metal-based coordination polymers because of their potential applications as functional solid materials in ion exchange, catalysis, and optical electronic and magnetic devices (Moulton & Zaworotko, 2001; Wen *et al.*, 2005). Pyridine-2,6-dicarboxylic acid N-oxide (pydco) shows limited steric hindrance and weak stacking interactions and can offer possibilities to form coordination polymers through a bridge formed by a carboxylate and N-oxide, which is a better electron donor than the ring N atom of pydco (Paul, 1984; Wen *et al.*, 2005)

The Cu1 atom in the title complex, (I), has a distorted square-pyramidal coordination geometry (Fig.1). The basal plane formed by two N atoms from the 2,2'-pyridine ligand and two O atoms from the pydco ligand. The apical position is occupied by a water molecule. A long distance [2.905 (3) Å] between Cu1 and O5($2-x$, $1-y$, $1-z$) at the other apical position indicates a very weak interaction. In the crystal strucrure, the intermolecular hydrogen bonds between the lattice water molecules and the coordination water molecule, and between the lattice water molecules and carboxylate O atoms form a sheet structure (Table 1). The sheets are linked by π···π interactions, forming a three dimensional supramolecular structure.

Experimental

Pydco(0.050 g, 8 mmol), $Cu(CH_3COO)_2$ (0.180 g, 12 mmol) and 2,2-pyridine (0.230 g, 15 mmol) were added in a mixed solvent of dry ethanol and acetonitrile. The mixture was heated for 5 h under reflux. During the process stirring and influx were required. The resultant was then filtered to give a pure solution which was infiltrated by diethyl ether freely in a closed vessel. After a week, single crystals of (I), suitable for X-Ray diffraction, were obtained.

Refinement

H atoms on water molecules were located in a difference Fourier map and fixed in the refinement with $U_{iso}(H) = 1.5U_{eq}(O)$. The other H atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The highest residual electron density was found 2.22Å from atom H2.

Figures

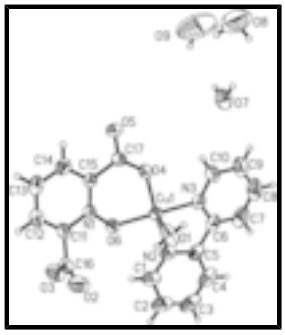


Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

Aqua[(pyridine-2,6-dicarboxylato-N-oxide-κ^2O^1,O^2) (2,2'-bipyridine-κ^2N,N')]copper(II) trihydrate

Crystal data

$[Cu(C_7H_3NO_5)(C_{10}H_8N_2)(H_2O)]\cdot 3H_2O$	$Z = 2$
$M_r = 472.89$	$F_{000} = 486$
Triclinic, $P\overline{1}$	$D_x = 1.563$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.8293$ (2) Å	Cell parameters from 3133 reflections
$b = 11.8827$ (4) Å	$\theta = 1.6$–26.0º
$c = 12.5673$ (4) Å	$\mu = 1.14$ mm^{-1}
$\alpha = 86.136$ (2)º	$T = 297$ (2) K
$\beta = 81.584$ (2)º	Block, blue
$\gamma = 86.151$ (2)º	0.20 × 0.18 × 0.10 mm
$V = 1004.87$ (6) Å^3	

Data collection

Bruker SMART APEX area-detector diffractometer	3953 independent reflections
Radiation source: fine-focus sealed tube	3133 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.031$
$T = 297(2)$ K	$\theta_{max} = 26.0$º
φ and ω scans	$\theta_{min} = 1.6$º
Absorption correction: multi-scan SADABS (Sheldrick, 1996)	$h = -8\rightarrow 8$
$T_{min} = 0.804$, $T_{max} = 0.894$	$k = -14\rightarrow 14$
13440 measured reflections	$l = -15\rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.140$

$S = 1.03$

3953 reflections

271 parameters

12 restraints

Primary atom site location: structure-invariant direct methods

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0811P)^2 + 0.7343P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 1.14$ e Å^{-3}

$\Delta\rho_{min} = -0.45$ e Å^{-3}

Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.73423 (8)	0.66865 (4)	0.54386 (4)	0.0387 (2)
N1	0.8064 (5)	0.5462 (3)	0.7427 (3)	0.0340 (8)
N2	0.7508 (6)	0.8345 (3)	0.5462 (3)	0.0434 (9)
N3	0.7073 (5)	0.7095 (3)	0.3898 (3)	0.0399 (8)
C1	0.7578 (8)	0.8909 (4)	0.6340 (5)	0.0544 (13)
H1	0.7615	0.8507	0.6998	0.065*
C2	0.7598 (8)	1.0069 (4)	0.6297 (6)	0.0673 (17)
H2	0.7645	1.0449	0.6916	0.081*
C3	0.7546 (8)	1.0655 (4)	0.5315 (6)	0.0687 (18)
H3	0.7577	1.1438	0.5264	0.082*
C4	0.7450 (8)	1.0084 (4)	0.4411 (5)	0.0613 (15)
H4	0.7398	1.0476	0.3748	0.074*
C5	0.7430 (7)	0.8909 (4)	0.4501 (4)	0.0464 (11)
C6	0.7264 (7)	0.8198 (4)	0.3611 (4)	0.0455 (11)
C7	0.7262 (9)	0.8595 (5)	0.2544 (5)	0.0705 (17)
H7	0.7398	0.9357	0.2347	0.085*
C8	0.7053 (10)	0.7840 (6)	0.1777 (5)	0.0756 (18)
H8	0.7048	0.8091	0.1060	0.091*
C9	0.6854 (8)	0.6722 (5)	0.2089 (4)	0.0621 (14)
H9	0.6705	0.6206	0.1587	0.074*
C10	0.6875 (7)	0.6371 (4)	0.3152 (4)	0.0483 (11)
H10	0.6749	0.5611	0.3360	0.058*
C11	0.7941 (7)	0.5565 (4)	0.8504 (3)	0.0403 (10)
C12	0.7898 (8)	0.4624 (4)	0.9192 (4)	0.0544 (13)
H12	0.7823	0.4699	0.9929	0.065*
C13	0.7967 (9)	0.3565 (4)	0.8801 (4)	0.0591 (14)
H13	0.7951	0.2922	0.9266	0.071*
C14	0.8059 (7)	0.3481 (4)	0.7708 (4)	0.0473 (11)
H14	0.8098	0.2770	0.7435	0.057*
C15	0.8096 (6)	0.4430 (3)	0.7008 (3)	0.0353 (9)
C16	0.7824 (8)	0.6755 (4)	0.8890 (3)	0.0460 (11)
C17	0.8104 (7)	0.4285 (3)	0.5819 (3)	0.0376 (9)
O1	0.4036 (5)	0.6858 (3)	0.6071 (3)	0.0617 (10)
H1A	0.3575	0.6869	0.6737	0.093*
H1B	0.3193	0.6594	0.5732	0.093*

O2	0.6125 (6)	0.7199 (3)	0.9079 (3)	0.0702 (11)
O3	0.9396 (6)	0.7143 (3)	0.9032 (3)	0.0634 (10)
O4	0.7598 (5)	0.5109 (2)	0.5205 (2)	0.0484 (8)
O5	0.8520 (5)	0.3325 (2)	0.5520 (3)	0.0514 (8)
O6	0.8213 (5)	0.6428 (2)	0.6828 (2)	0.0472 (8)
O7	0.6884 (6)	0.3775 (3)	0.1792 (3)	0.0639 (10)
H7A	0.8030	0.3448	0.1597	0.096*
H7B	0.6013	0.3375	0.1596	0.096*
O8	0.5451 (13)	0.0649 (6)	0.0755 (9)	0.199 (4)
H8A	0.4633	0.1262	0.0799	0.299*
H8B	0.6187	0.0809	0.1323	0.299*
O9	0.9391 (12)	0.0685 (6)	0.0931 (9)	0.193 (4)
H9B	0.9337	0.0859	0.0239	0.290*
H9A	0.9730	0.1342	0.1132	0.290*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0540 (4)	0.0304 (3)	0.0327 (3)	−0.0019 (2)	−0.0102 (2)	0.00015 (19)
N1	0.037 (2)	0.0344 (17)	0.0310 (18)	−0.0007 (14)	−0.0097 (14)	0.0005 (13)
N2	0.043 (2)	0.0346 (18)	0.052 (2)	−0.0025 (16)	−0.0064 (17)	−0.0023 (16)
N3	0.037 (2)	0.043 (2)	0.039 (2)	0.0019 (15)	−0.0065 (16)	0.0039 (15)
C1	0.058 (3)	0.039 (2)	0.066 (3)	−0.003 (2)	−0.006 (3)	−0.011 (2)
C2	0.056 (3)	0.046 (3)	0.104 (5)	−0.001 (2)	−0.012 (3)	−0.028 (3)
C3	0.049 (3)	0.033 (2)	0.123 (6)	−0.002 (2)	−0.012 (3)	0.001 (3)
C4	0.042 (3)	0.042 (3)	0.097 (5)	−0.001 (2)	−0.011 (3)	0.018 (3)
C5	0.034 (3)	0.040 (2)	0.063 (3)	−0.0007 (19)	−0.005 (2)	0.007 (2)
C6	0.039 (3)	0.047 (3)	0.047 (3)	0.0040 (19)	−0.004 (2)	0.011 (2)
C7	0.073 (4)	0.066 (4)	0.065 (4)	0.003 (3)	−0.002 (3)	0.028 (3)
C8	0.084 (5)	0.100 (5)	0.038 (3)	0.007 (4)	−0.008 (3)	0.013 (3)
C9	0.059 (4)	0.088 (4)	0.040 (3)	0.000 (3)	−0.013 (2)	−0.003 (3)
C10	0.047 (3)	0.060 (3)	0.039 (3)	0.001 (2)	−0.012 (2)	−0.003 (2)
C11	0.039 (3)	0.048 (2)	0.034 (2)	0.0001 (19)	−0.0069 (18)	−0.0038 (18)
C12	0.068 (4)	0.062 (3)	0.034 (2)	−0.002 (3)	−0.011 (2)	0.005 (2)
C13	0.075 (4)	0.054 (3)	0.047 (3)	−0.003 (3)	−0.014 (3)	0.016 (2)
C14	0.054 (3)	0.039 (2)	0.048 (3)	−0.002 (2)	−0.008 (2)	0.0042 (19)
C15	0.035 (2)	0.035 (2)	0.036 (2)	0.0003 (17)	−0.0075 (17)	0.0010 (16)
C16	0.054 (3)	0.055 (3)	0.030 (2)	0.001 (2)	−0.011 (2)	−0.0052 (19)
C17	0.040 (2)	0.034 (2)	0.040 (2)	−0.0056 (17)	−0.0072 (19)	−0.0037 (17)
O1	0.057 (2)	0.091 (3)	0.0394 (19)	−0.022 (2)	−0.0063 (16)	−0.0050 (17)
O2	0.059 (3)	0.072 (2)	0.084 (3)	0.011 (2)	−0.017 (2)	−0.032 (2)
O3	0.060 (2)	0.067 (2)	0.069 (2)	−0.0102 (19)	−0.0181 (19)	−0.0174 (19)
O4	0.077 (2)	0.0356 (16)	0.0361 (17)	0.0006 (15)	−0.0208 (16)	−0.0035 (13)
O5	0.076 (2)	0.0313 (16)	0.0492 (19)	0.0016 (15)	−0.0160 (17)	−0.0059 (13)
O6	0.076 (2)	0.0322 (15)	0.0362 (17)	−0.0070 (15)	−0.0180 (15)	0.0010 (12)
O7	0.060 (2)	0.082 (3)	0.049 (2)	−0.006 (2)	−0.0057 (17)	−0.0002 (18)
O8	0.183 (8)	0.102 (5)	0.341 (13)	0.055 (5)	−0.136 (8)	−0.074 (6)
O9	0.153 (7)	0.092 (5)	0.327 (13)	−0.040 (5)	0.007 (7)	−0.019 (6)

Geometric parameters (Å, °)

Cu1—O4	1.910 (3)	C8—H8	0.9300
Cu1—O6	1.925 (3)	C9—C10	1.374 (7)
Cu1—N2	1.984 (4)	C9—H9	0.9300
Cu1—N3	1.996 (4)	C10—H10	0.9300
Cu1—O1	2.282 (4)	C11—C12	1.366 (6)
N1—O6	1.331 (4)	C11—C16	1.518 (6)
N1—C11	1.357 (5)	C12—C13	1.376 (7)
N1—C15	1.365 (5)	C12—H12	0.9300
N2—C1	1.336 (6)	C13—C14	1.376 (7)
N2—C5	1.348 (6)	C13—H13	0.9300
N3—C10	1.340 (6)	C14—C15	1.382 (6)
N3—C6	1.345 (6)	C14—H14	0.9300
C1—C2	1.376 (7)	C15—C17	1.515 (6)
C1—H1	0.9300	C16—O3	1.237 (6)
C2—C3	1.380 (9)	C16—O2	1.239 (6)
C2—H2	0.9300	C17—O5	1.227 (5)
C3—C4	1.373 (9)	C17—O4	1.266 (5)
C3—H3	0.9300	O1—H1A	0.85
C4—C5	1.394 (7)	O1—H1B	0.85
C4—H4	0.9300	O7—H7A	0.86
C5—C6	1.468 (7)	O7—H7B	0.86
C6—C7	1.391 (7)	O8—H8A	0.89
C7—C8	1.389 (9)	O8—H8B	0.97
C7—H7	0.9300	O9—H9B	0.89
C8—C9	1.370 (9)	O9—H9A	0.89
O4—Cu1—O6	91.68 (12)	C6—C7—H7	120.4
O4—Cu1—N2	169.20 (16)	C9—C8—C7	119.1 (5)
O6—Cu1—N2	92.21 (14)	C9—C8—H8	120.4
O4—Cu1—N3	92.26 (14)	C7—C8—H8	120.4
O6—Cu1—N3	166.45 (15)	C8—C9—C10	119.4 (6)
N2—Cu1—N3	81.76 (15)	C8—C9—H9	120.3
O4—Cu1—O1	99.39 (15)	C10—C9—H9	120.3
O6—Cu1—O1	96.04 (14)	N3—C10—C9	121.8 (5)
N2—Cu1—O1	90.21 (15)	N3—C10—H10	119.1
N3—Cu1—O1	96.10 (14)	C9—C10—H10	119.1
O6—N1—C11	115.0 (3)	N1—C11—C12	120.1 (4)
O6—N1—C15	123.6 (3)	N1—C11—C16	117.1 (4)
C11—N1—C15	121.4 (4)	C12—C11—C16	122.8 (4)
C1—N2—C5	120.2 (4)	C11—C12—C13	120.4 (5)
C1—N2—Cu1	125.4 (3)	C11—C12—H12	119.8
C5—N2—Cu1	114.2 (3)	C13—C12—H12	119.8
C10—N3—C6	119.9 (4)	C12—C13—C14	118.5 (4)
C10—N3—Cu1	126.0 (3)	C12—C13—H13	120.7
C6—N3—Cu1	113.9 (3)	C14—C13—H13	120.7
N2—C1—C2	121.9 (5)	C13—C14—C15	121.4 (5)
N2—C1—H1	119.0	C13—C14—H14	119.3

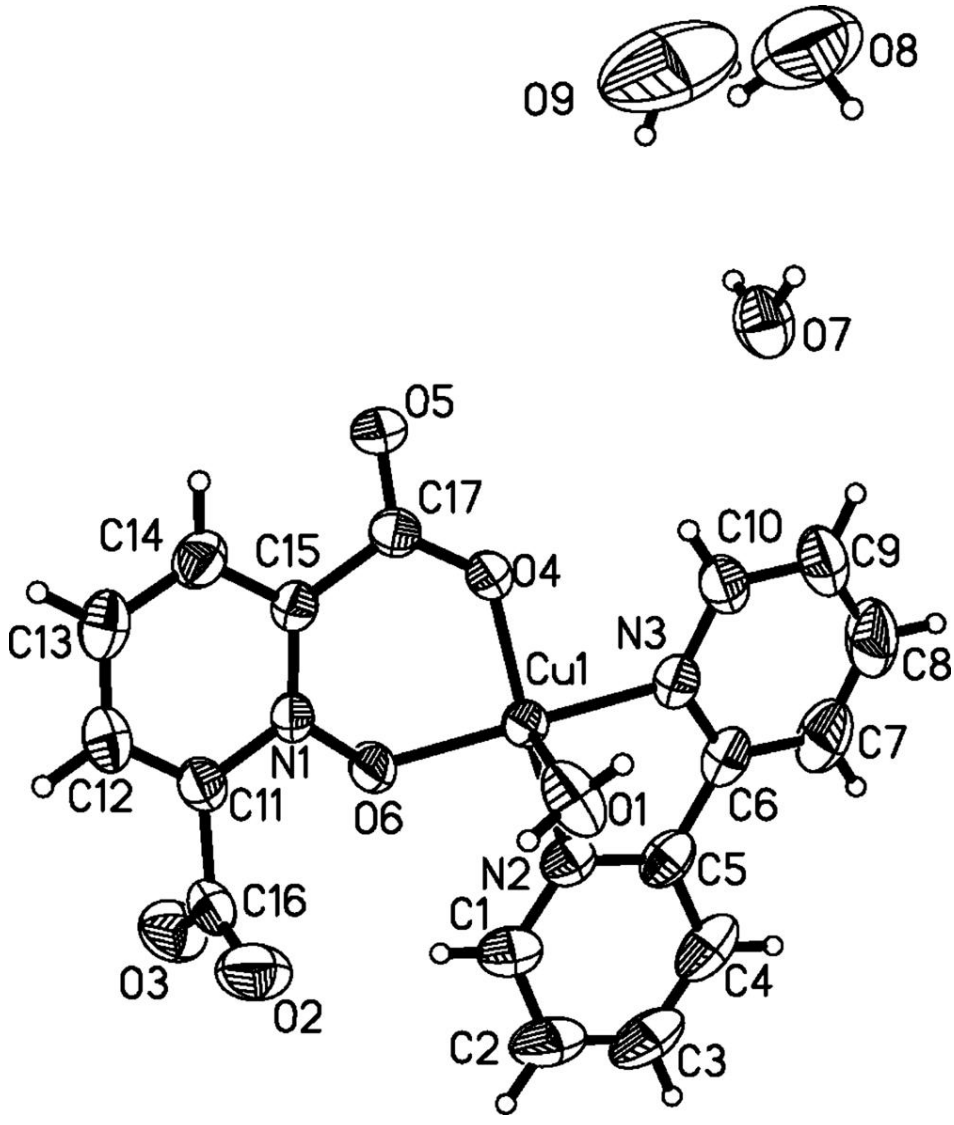
C2—C1—H1	119.0	C15—C14—H14	119.3
C1—C2—C3	118.4 (6)	N1—C15—C14	118.2 (4)
C1—C2—H2	120.8	N1—C15—C17	122.9 (3)
C3—C2—H2	120.8	C14—C15—C17	118.9 (4)
C4—C3—C2	120.2 (5)	O3—C16—O2	127.6 (5)
C4—C3—H3	119.9	O3—C16—C11	117.2 (4)
C2—C3—H3	119.9	O2—C16—C11	115.0 (4)
C3—C4—C5	119.0 (5)	O5—C17—O4	123.5 (4)
C3—C4—H4	120.5	O5—C17—C15	115.9 (4)
C5—C4—H4	120.5	O4—C17—C15	120.5 (4)
N2—C5—C4	120.3 (5)	Cu1—O1—H1A	123.5
N2—C5—C6	115.0 (4)	Cu1—O1—H1B	120.5
C4—C5—C6	124.7 (5)	H1A—O1—H1B	109.6
N3—C6—C7	120.5 (5)	C17—O4—Cu1	129.6 (3)
N3—C6—C5	114.9 (4)	N1—O6—Cu1	124.5 (2)
C7—C6—C5	124.6 (5)	H7A—O7—H7B	107.6
C8—C7—C6	119.3 (5)	H8A—O8—H8B	96.9
C8—C7—H7	120.4	H9B—O9—H9A	100.1

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···O7^{i}	0.85	1.94	2.733 (5)	154
O1—H1B···O5^{i}	0.85	2.09	2.867 (5)	152
O1—H1B···O4^{i}	0.85	2.53	3.265 (5)	144
O7—H7A···O3^{ii}	0.86	1.93	2.780 (6)	171
O7—H7B···O2^{i}	0.86	1.97	2.813 (5)	166
O8—H8A···O2^{i}	0.89	1.88	2.716 (8)	157
O8—H8B···O9	0.97	2.17	2.735 (12)	116
O9—H9A···O3^{ii}	0.89	1.92	2.768 (7)	158
O9—H9B···O9^{iii}	0.89	2.46	2.930 (19)	113

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+2, −*y*+1, −*z*+1; (iii) −*x*+2, −*y*, −*z*.

Fig. 1


O9
O8
O7
O5
C17
C14
C15
O4
C10
C9
C13
N3
C8
Cu1
N1
C7
C12
C11
O6
O1
C6
N2
C5
C16
C1
O3
C4
O2
C2
C3