

## Aquabis(3-chlorobenzoato- $\kappa$ O)bis(*N,N*-diethylnicotinamide- $\kappa$ N)copper(II)

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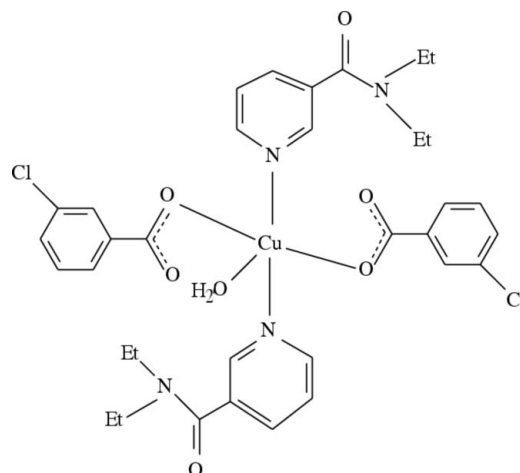
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.032;  $wR$  factor = 0.086; data-to-parameter ratio = 27.8.

The title compound,  $[\text{Cu}(\text{C}_7\text{H}_4\text{ClO}_2)_2(\text{C}_{10}\text{H}_{14}\text{N}_2\text{O})_2(\text{H}_2\text{O})]$ , has twofold symmetry with the  $\text{Cu}^{\text{II}}$  cation and the O atom of the coordinating water molecule located on the axis. The  $\text{Cu}^{\text{II}}$  cation is coordinated by two carboxylate O atoms of chlorobenzoate (CB) anions, two N atoms of *N,N*-diethylnicotinamide (DNA) ligands and one water molecule in a distorted  $\text{N}_2\text{O}_3$  square-pyramidal geometry. The benzene and pyridine rings are oriented at a dihedral angle of  $82.51(6)^\circ$ . In the anionic ligand, the carboxylate group is twisted away from the attached benzene ring by  $12.85(11)^\circ$ . In the crystal,  $\text{O} \cdots \text{H} \cdots \text{O}$  hydrogen bonds between the coordinating water molecule and the carboxyl group link the complex molecules into supramolecular chains running along the  $c$ -axis direction.

### Related literature

For literature on niacin, see: Krishnamachari (1974). For information on the nicotinic acid derivative *N,N*-diethylnicotinamide, see: Bigoli *et al.* (1972). For related structures, see: Hökelek *et al.* (1996, 2009*a,b*); Hökelek & Necefoğlu (1998, 2007); Necefoğlu *et al.* (2011*a,b,c*). For bond-length data, see: Allen *et al.* (1987).



### Experimental

#### Crystal data

$[\text{Cu}(\text{C}_7\text{H}_4\text{ClO}_2)_2(\text{C}_{10}\text{H}_{14}\text{N}_2\text{O})_2(\text{H}_2\text{O})]$   
 $M_r = 749.13$   
 Orthorhombic, *Iba*2  
 $a = 15.9185(9)$  Å  
 $b = 19.2366(11)$  Å  
 $c = 11.5535(7)$  Å

$V = 3537.9(4)$  Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.82$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.35 \times 0.20 \times 0.15$  mm

#### Data collection

Bruker SMART BREEZE CCD diffractometer  
 Absorption correction: multi-scan (*SADABS*; Bruker, 2012)  
 $T_{\text{min}} = 0.821$ ,  $T_{\text{max}} = 0.884$

75834 measured reflections  
 6232 independent reflections  
 5189 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.048$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.032$   
 $wR(F^2) = 0.086$   
 $S = 1.06$   
 6232 reflections  
 224 parameters  
 1 restraint

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.25$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.39$  e Å<sup>-3</sup>  
 Absolute structure: Flack (1983), with no Friedel pairs measured  
 Flack parameter: 0.027 (10)

**Table 1**

Selected bond lengths (Å).

Cu1—N1	2.0294 (12)	Cu1—O4	2.238 (2)
Cu1—O1	1.9337 (10)		

**Table 2**

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O4}-\text{H41} \cdots \text{O2}^i$	0.79 (4)	1.95 (3)	2.7367 (17)	171 (4)

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

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINTE* (Bruker, 2012); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics:

ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5719).

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## supporting information

*Acta Cryst.* (2013). E69, m458–m459 [doi:10.1107/S1600536813018989]

**Aquabis(3-chlorobenzoato- $\kappa$ O)bis(*N,N*-diethylnicotinamide- $\kappa$ N)copper(II)****Nihat Bozkurt, Tuncay Tunç, Nagihan Çaylak Delibaş, Hacali Necefoğlu and Tuncer Hökelek****S1. Comment**

As a part of our ongoing investigations of transition metal complexes of nicotinamide (NA), one form of niacin (Krishnamachari, 1974), and/or the nicotinic acid derivative *N,N*-diethylnicotinamide (DNA), an important respiratory stimulant (Bigoli *et al.*, 1972), the title compound was synthesized and its crystal structure is reported herein.

The asymmetric unit of the title mononuclear Cu<sup>II</sup> complex, (Fig. 1), contains one-half molecule, the Cu<sup>II</sup> cation is located on a twofold rotation axis and is coordinated by carboxylate O atoms of two chlorobenzoate (CB) anions, N atoms of two *N,N*-diethylnicotinamide (DNA) ligands and by one water molecule, located on a twofold rotation axis, all ligands coordinating in a monodentate manner. The crystal structures of similar complexes of Cu<sup>II</sup>, Co<sup>II</sup>, Ni<sup>II</sup>, Mn<sup>II</sup> and Zn<sup>II</sup> cations, [Cu(C<sub>7</sub>H<sub>5</sub>O<sub>2</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>14</sub>N<sub>2</sub>O)<sub>2</sub>] (Hökelek *et al.*, 1996); [Cu(C<sub>9</sub>H<sub>9</sub>O<sub>2</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>14</sub>N<sub>2</sub>O)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] (Necefoğlu *et al.*, 2011*a*); [Cu(C<sub>7</sub>H<sub>4</sub>FO<sub>2</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>14</sub>N<sub>2</sub>O)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] (Necefoğlu *et al.*, 2011*b*); [Co(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)<sub>2</sub>(C<sub>7</sub>H<sub>4</sub>NO<sub>4</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] (Hökelek & Necefoğlu, 1998); [Co(C<sub>9</sub>H<sub>9</sub>O<sub>2</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>14</sub>N<sub>2</sub>O)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] (Necefoğlu *et al.*, 2011*c*); [Ni(C<sub>7</sub>H<sub>4</sub>ClO<sub>2</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] (Hökelek *et al.*, 2009*a*); [Mn(C<sub>9</sub>H<sub>10</sub>NO<sub>2</sub>)<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>].2H<sub>2</sub>O (Hökelek & Necefoğlu, 2007) and [Zn(C<sub>7</sub>H<sub>4</sub>BrO<sub>2</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>6</sub>N<sub>2</sub>O)<sub>2</sub>(H<sub>2</sub>O)<sub>2</sub>] (Hökelek *et al.*, 2009*b*) have also been reported. In the first copper(II) complex mentioned above the two benzoate ions coordinate to the Cu<sup>II</sup> atom as bidentate ligands, while in the other structures all the ligands coordinate in a monodentate manner.

In the title complex, the four symmetry related O and N atoms (O1, O1a and N1, N1a) [symmetry code: (a) - *x*, 1 - *y*, *z*] in the equatorial plane around the Cu<sup>II</sup> cation form a distorted square-planar arrangement, while the distorted square-pyramidal coordination is completed by the water O atom (O4) in the axial position.

The Cu—O bond lengths are 1.9346 (11) Å (for benzoate oxygen) and 2.238 (2) Å (for water oxygen), and the Cu—N bond length is 2.0303 (14) Å, close to standard values (Allen *et al.*, 1987). The Cu atom is displaced out of the mean-plane of the carboxylate group (O1/C1/O2) by -0.1606 (1) Å. The dihedral angle between the planar carboxylate group and the adjacent benzene ring *A* (C2—C7) is 12.85 (11)°. The benzene *A* (C2—C7) and the pyridine *B* (N1/C8—C12) rings are oriented at a dihedral angle of 82.51 (6)°.

In the crystal, strong O—H...O hydrogen bonds (Table 2) link the water hydrogens to the carboxylate oxygens into infinite chains along the *c*-axis.

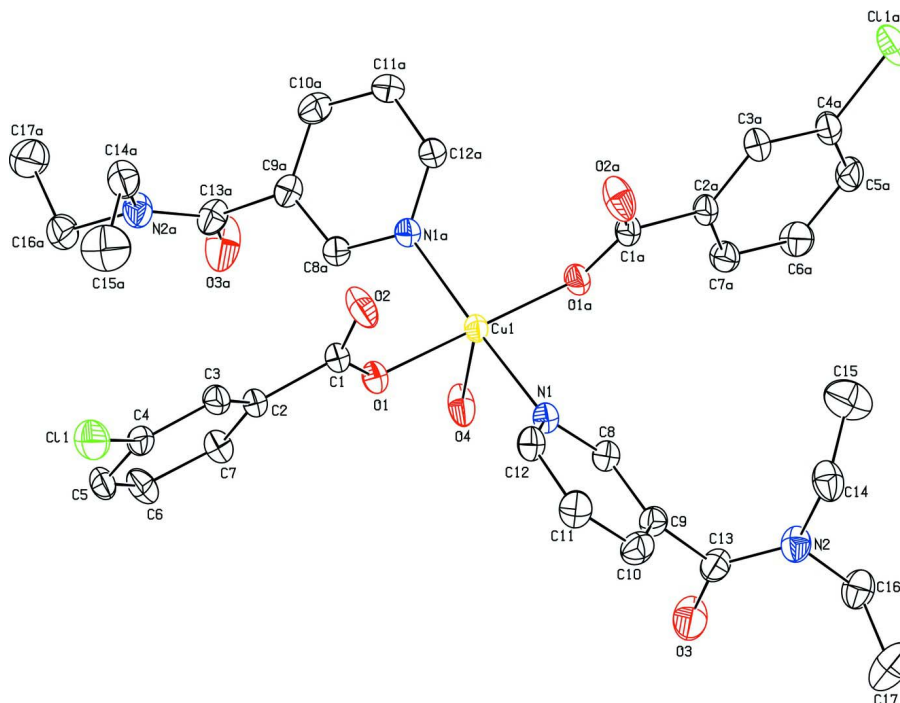
**S2. Experimental**

The title compound was prepared by the reaction of CuSO<sub>4</sub>·5H<sub>2</sub>O (1.25 g, 5 mmol) in H<sub>2</sub>O (100 ml) and diethylnicotinamide (1.78 g, 10 mmol) in H<sub>2</sub>O (20 ml) with sodium 3-chlorobenzoate (1.79 g, 10 mmol) in H<sub>2</sub>O (100 ml). The mixture was set aside to crystallize at ambient temperature for five days, giving blue single crystals.

**S3. Refinement**

Atom H41 (for H<sub>2</sub>O) was located in a difference Fourier map and was refined freely. The C-bound H-atoms were positioned geometrically with C—H = 0.93, 0.97 and 0.96 Å, for aromatic, methylene and methyl H-atoms, respectively,

and constrained to ride on their parent atoms, with  $U_{\text{iso}}(\text{H}) = k \times U_{\text{eq}}(\text{C})$ , where  $k = 1.5$  for methyl H-atoms and  $k = 1.2$  for all other H-atoms.



**Figure 1**

The molecular structure of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level [symmetry code: (a)  $2 - x, 1 - y, z$ ]. Hydrogen atoms have been omitted for clarity.

### Aquabis(3-chlorobenzoato- $\kappa$ O)bis(*N,N*-diethylnicotinamide- $\kappa$ N)copper(II)

#### Crystal data

$[\text{Cu}(\text{C}_7\text{H}_4\text{ClO}_2)_2(\text{C}_{10}\text{H}_{14}\text{N}_2\text{O})_2(\text{H}_2\text{O})]$

$M_r = 749.13$

Orthorhombic, *Iba*2

Hall symbol: I 2 -2c

$a = 15.9185$  (9) Å

$b = 19.2366$  (11) Å

$c = 11.5535$  (7) Å

$V = 3537.9$  (4) Å<sup>3</sup>

$Z = 4$

$F(000) = 1556$

$D_x = 1.406$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 9278 reflections

$\theta = 2.2\text{--}30.7^\circ$

$\mu = 0.82$  mm<sup>-1</sup>

$T = 296$  K

Block, blue

$0.35 \times 0.20 \times 0.15$  mm

#### Data collection

Bruker SMART BREEZE CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2012)

$T_{\text{min}} = 0.821$ ,  $T_{\text{max}} = 0.884$

75834 measured reflections

6232 independent reflections

5189 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.048$

$\theta_{\text{max}} = 32.3^\circ$ ,  $\theta_{\text{min}} = 1.7^\circ$

$h = -23 \rightarrow 23$

$k = -28 \rightarrow 28$

$l = -17 \rightarrow 16$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.086$

$S = 1.06$

6232 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

$w = 1/[\sigma^2(F_o^2) + (0.049P)^2 + 0.4582P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.39 \text{ e } \text{\AA}^{-3}$

Absolute structure: Flack (1983), with no  
Friedel pairs measured

Absolute structure parameter: 0.027 (10)

*Special details*

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu1	1.0000	0.5000	0.41873 (3)	0.03000 (6)
Cl1	1.46828 (3)	0.59979 (3)	0.63513 (5)	0.06445 (15)
O1	1.11786 (6)	0.52434 (6)	0.41852 (13)	0.0393 (2)
O2	1.14688 (8)	0.52348 (10)	0.60832 (13)	0.0614 (4)
O3	0.84439 (11)	0.72659 (11)	0.16031 (16)	0.0800 (5)
O4	1.0000	0.5000	0.22505 (18)	0.0538 (6)
H41	1.0395 (19)	0.4937 (15)	0.185 (4)	0.083 (10)*
N1	0.96843 (7)	0.60210 (6)	0.42656 (14)	0.0353 (2)
N2	0.74305 (10)	0.72799 (9)	0.29566 (14)	0.0469 (3)
C1	1.16668 (9)	0.52875 (9)	0.50510 (15)	0.0363 (3)
C2	1.25768 (9)	0.54254 (8)	0.47400 (14)	0.0349 (3)
C3	1.31396 (9)	0.56202 (9)	0.56019 (15)	0.0391 (3)
H3	1.2964	0.5673	0.6364	0.047*
C4	1.39704 (10)	0.57329 (8)	0.52927 (16)	0.0414 (3)
C5	1.42506 (9)	0.56541 (8)	0.4176 (2)	0.0469 (3)
H5	1.4812	0.5729	0.3992	0.056*
C6	1.36773 (12)	0.54600 (10)	0.33236 (19)	0.0514 (4)
H6	1.3857	0.5403	0.2563	0.062*
C7	1.28426 (11)	0.53510 (9)	0.36030 (17)	0.0422 (4)
H7	1.2460	0.5228	0.3030	0.051*
C8	0.90811 (10)	0.62540 (8)	0.35534 (15)	0.0372 (3)
H8	0.8802	0.5936	0.3084	0.045*
C9	0.88569 (10)	0.69476 (8)	0.34881 (14)	0.0398 (3)

C10	0.92796 (12)	0.74174 (8)	0.4190 (2)	0.0510 (4)
H10	0.9146	0.7888	0.4161	0.061*
C11	0.99002 (12)	0.71828 (10)	0.4931 (2)	0.0509 (4)
H11	1.0188	0.7490	0.5409	0.061*
C12	1.00827 (10)	0.64755 (10)	0.49440 (18)	0.0412 (3)
H12	1.0497	0.6314	0.5443	0.049*
C13	0.82193 (13)	0.71794 (10)	0.26044 (15)	0.0462 (4)
C14	0.71426 (12)	0.71772 (12)	0.4142 (2)	0.0551 (4)
H14A	0.6784	0.7563	0.4361	0.066*
H14B	0.7625	0.7177	0.4656	0.066*
C15	0.6667 (2)	0.65112 (17)	0.4298 (3)	0.0907 (8)
H15A	0.6454	0.6486	0.5074	0.136*
H15B	0.7036	0.6125	0.4159	0.136*
H15C	0.6208	0.6496	0.3760	0.136*
C16	0.68061 (14)	0.75374 (11)	0.2115 (2)	0.0546 (5)
H16A	0.6255	0.7363	0.2325	0.066*
H16B	0.6943	0.7358	0.1353	0.066*
C17	0.6778 (2)	0.83180 (13)	0.2067 (3)	0.0864 (9)
H17A	0.6405	0.8462	0.1461	0.130*
H17B	0.7332	0.8495	0.1916	0.130*
H17C	0.6581	0.8496	0.2795	0.130*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Cu1	0.02380 (9)	0.03532 (10)	0.03087 (11)	0.00119 (8)	0.000	0.000
Cl1	0.0404 (2)	0.0777 (3)	0.0752 (4)	-0.0116 (2)	-0.0184 (2)	0.0065 (3)
O1	0.0265 (4)	0.0465 (5)	0.0449 (5)	-0.0019 (4)	-0.0005 (5)	-0.0018 (7)
O2	0.0334 (6)	0.1087 (12)	0.0420 (7)	-0.0084 (7)	0.0062 (5)	0.0074 (8)
O3	0.0700 (10)	0.1258 (14)	0.0442 (8)	0.0380 (10)	0.0141 (8)	0.0253 (10)
O4	0.0329 (9)	0.1020 (18)	0.0265 (10)	0.0133 (9)	0.000	0.000
N1	0.0290 (5)	0.0383 (5)	0.0385 (7)	0.0010 (4)	0.0002 (6)	-0.0028 (6)
N2	0.0421 (7)	0.0617 (8)	0.0369 (7)	0.0086 (6)	-0.0025 (6)	-0.0013 (7)
C1	0.0271 (6)	0.0404 (8)	0.0414 (8)	0.0011 (5)	0.0034 (5)	0.0022 (6)
C2	0.0259 (6)	0.0380 (7)	0.0408 (8)	0.0008 (5)	0.0023 (6)	0.0036 (6)
C3	0.0304 (6)	0.0462 (8)	0.0409 (8)	-0.0006 (6)	-0.0011 (6)	0.0060 (6)
C4	0.0292 (6)	0.0394 (7)	0.0556 (10)	-0.0005 (6)	-0.0050 (6)	0.0073 (7)
C5	0.0283 (6)	0.0440 (7)	0.0683 (10)	-0.0026 (5)	0.0105 (8)	0.0012 (10)
C6	0.0425 (9)	0.0593 (10)	0.0523 (11)	-0.0073 (7)	0.0181 (8)	-0.0060 (8)
C7	0.0353 (7)	0.0488 (9)	0.0426 (9)	-0.0033 (6)	0.0052 (7)	-0.0042 (7)
C8	0.0340 (7)	0.0392 (7)	0.0385 (8)	0.0023 (5)	-0.0017 (6)	-0.0016 (6)
C9	0.0390 (7)	0.0417 (7)	0.0389 (8)	0.0065 (6)	0.0043 (6)	0.0030 (6)
C10	0.0548 (9)	0.0352 (6)	0.0631 (10)	0.0041 (6)	0.0000 (11)	-0.0029 (10)
C11	0.0458 (9)	0.0426 (9)	0.0643 (12)	-0.0027 (7)	-0.0054 (8)	-0.0141 (8)
C12	0.0339 (7)	0.0463 (8)	0.0435 (9)	0.0026 (6)	-0.0029 (6)	-0.0071 (7)
C13	0.0503 (9)	0.0505 (9)	0.0377 (9)	0.0151 (8)	0.0031 (7)	0.0035 (7)
C14	0.0438 (8)	0.0812 (12)	0.0405 (8)	0.0058 (8)	0.0010 (9)	-0.0060 (12)
C15	0.099 (2)	0.0903 (18)	0.0825 (19)	-0.0149 (15)	0.0178 (19)	0.0077 (18)

C16	0.0501 (10)	0.0630 (11)	0.0508 (10)	0.0082 (8)	-0.0108 (8)	-0.0045 (9)
C17	0.094 (2)	0.0615 (13)	0.104 (2)	0.0226 (13)	-0.0383 (17)	-0.0114 (14)

*Geometric parameters (Å, °)*

Cu1—N1	2.0294 (12)	C7—H7	0.9300
Cu1—N1 <sup>i</sup>	2.0294 (12)	C8—H8	0.9300
Cu1—O1	1.9337 (10)	C9—C8	1.383 (2)
Cu1—O1 <sup>i</sup>	1.9337 (10)	C9—C10	1.388 (3)
Cu1—O4	2.238 (2)	C9—C13	1.507 (2)
Cl1—C4	1.7439 (18)	C10—C11	1.383 (3)
O1—C1	1.269 (2)	C10—H10	0.9300
O4—H41	0.79 (4)	C11—H11	0.9300
N1—C8	1.341 (2)	C12—C11	1.391 (3)
N1—C12	1.335 (2)	C12—H12	0.9300
N2—C13	1.334 (2)	C13—O3	1.222 (2)
N2—C14	1.458 (3)	C14—C15	1.499 (4)
N2—C16	1.476 (3)	C14—H14A	0.9700
C1—O2	1.238 (2)	C14—H14B	0.9700
C1—C2	1.516 (2)	C15—H15A	0.9600
C2—C3	1.391 (2)	C15—H15B	0.9600
C2—C7	1.387 (2)	C15—H15C	0.9600
C3—C4	1.387 (2)	C16—C17	1.503 (3)
C3—H3	0.9300	C16—H16A	0.9700
C5—C4	1.373 (3)	C16—H16B	0.9700
C5—C6	1.394 (3)	C17—H17A	0.9600
C5—H5	0.9300	C17—H17B	0.9600
C6—H6	0.9300	C17—H17C	0.9600
C7—C6	1.383 (2)		
O1—Cu1—O1 <sup>i</sup>	179.86 (9)	C9—C8—H8	118.6
O1—Cu1—O4	89.93 (5)	C8—C9—C10	118.11 (16)
O1 <sup>i</sup> —Cu1—O4	89.93 (5)	C8—C9—C13	119.75 (16)
O1—Cu1—N1	90.35 (5)	C10—C9—C13	121.96 (15)
O1 <sup>i</sup> —Cu1—N1	89.66 (5)	C9—C10—H10	120.2
O1—Cu1—N1 <sup>i</sup>	89.66 (5)	C11—C10—C9	119.69 (15)
O1 <sup>i</sup> —Cu1—N1 <sup>i</sup>	90.35 (5)	C11—C10—H10	120.2
N1—Cu1—O4	92.55 (5)	C10—C11—C12	118.36 (17)
N1 <sup>i</sup> —Cu1—O4	92.55 (5)	C10—C11—H11	120.8
N1 <sup>i</sup> —Cu1—N1	174.89 (9)	C12—C11—H11	120.8
C1—O1—Cu1	127.53 (12)	N1—C12—C11	122.34 (17)
Cu1—O4—H41	126 (3)	N1—C12—H12	118.8
C8—N1—Cu1	118.25 (11)	C11—C12—H12	118.8
C12—N1—Cu1	122.85 (12)	O3—C13—N2	122.97 (18)
C12—N1—C8	118.79 (13)	O3—C13—C9	118.98 (17)
C13—N2—C14	124.25 (16)	N2—C13—C9	118.05 (16)
C13—N2—C16	118.78 (16)	N2—C14—C15	112.8 (2)
C14—N2—C16	116.93 (15)	N2—C14—H14A	109.0

O1—C1—C2	114.20 (14)	N2—C14—H14B	109.0
O2—C1—O1	126.72 (15)	C15—C14—H14A	109.0
O2—C1—C2	119.08 (15)	C15—C14—H14B	109.0
C3—C2—C1	119.53 (15)	H14A—C14—H14B	107.8
C7—C2—C1	119.85 (14)	C14—C15—H15A	109.5
C7—C2—C3	120.62 (14)	C14—C15—H15B	109.5
C2—C3—H3	120.9	C14—C15—H15C	109.5
C4—C3—C2	118.15 (16)	H15A—C15—H15B	109.5
C4—C3—H3	120.9	H15A—C15—H15C	109.5
C3—C4—C11	119.02 (14)	H15B—C15—H15C	109.5
C5—C4—C11	118.68 (12)	N2—C16—C17	112.26 (19)
C5—C4—C3	122.30 (16)	N2—C16—H16A	109.2
C4—C5—C6	118.74 (14)	N2—C16—H16B	109.2
C4—C5—H5	120.6	C17—C16—H16A	109.2
C6—C5—H5	120.6	C17—C16—H16B	109.2
C5—C6—H6	119.8	H16A—C16—H16B	107.9
C7—C6—C5	120.31 (18)	C16—C17—H17A	109.5
C7—C6—H6	119.8	C16—C17—H17B	109.5
C2—C7—H7	120.1	C16—C17—H17C	109.5
C6—C7—C2	119.87 (17)	H17A—C17—H17B	109.5
C6—C7—H7	120.1	H17A—C17—H17C	109.5
N1—C8—C9	122.71 (15)	H17B—C17—H17C	109.5
N1—C8—H8	118.6		
O4—Cu1—O1—C1	-173.97 (13)	O1—C1—C2—C7	-13.2 (2)
N1—Cu1—O1—C1	93.48 (14)	O2—C1—C2—C3	-12.1 (2)
N1 <sup>i</sup> —Cu1—O1—C1	-81.41 (14)	O2—C1—C2—C7	167.34 (19)
O1—Cu1—N1—C8	133.82 (13)	C1—C2—C3—C4	179.08 (15)
O1 <sup>i</sup> —Cu1—N1—C8	-46.04 (13)	C7—C2—C3—C4	-0.4 (2)
O1—Cu1—N1—C12	-42.21 (15)	C1—C2—C7—C6	-178.40 (16)
O1 <sup>i</sup> —Cu1—N1—C12	137.94 (15)	C3—C2—C7—C6	1.1 (3)
O4—Cu1—N1—C8	43.87 (12)	C2—C3—C4—C11	178.47 (12)
O4—Cu1—N1—C12	-132.15 (14)	C2—C3—C4—C5	-0.4 (2)
Cu1—O1—C1—O2	-6.0 (3)	C6—C5—C4—C11	-178.34 (14)
Cu1—O1—C1—C2	174.59 (9)	C6—C5—C4—C3	0.6 (3)
Cu1—N1—C8—C9	-175.86 (13)	C4—C5—C6—C7	0.1 (3)
C12—N1—C8—C9	0.3 (3)	C2—C7—C6—C5	-0.9 (3)
Cu1—N1—C12—C11	175.38 (16)	C10—C9—C8—N1	0.2 (3)
C8—N1—C12—C11	-0.6 (3)	C13—C9—C8—N1	175.42 (16)
C14—N2—C13—O3	179.6 (2)	C8—C9—C10—C11	-0.5 (3)
C14—N2—C13—C9	-0.3 (3)	C13—C9—C10—C11	-175.56 (19)
C16—N2—C13—O3	-2.8 (3)	C8—C9—C13—O3	-79.9 (3)
C16—N2—C13—C9	177.28 (16)	C8—C9—C13—N2	100.0 (2)
C13—N2—C14—C15	-102.9 (3)	C10—C9—C13—O3	95.1 (3)
C16—N2—C14—C15	79.5 (3)	C10—C9—C13—N2	-85.0 (2)
C13—N2—C16—C17	-88.5 (3)	C9—C10—C11—C12	0.2 (3)



C14—N2—C16—C17	89.2 (3)	N1—C12—C11—C10	0.4 (3)
O1—C1—C2—C3	167.34 (15)		

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

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

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
O4—H41 $\cdots$ O2 <sup>ii</sup>	0.79 (4)	1.95 (3)	2.7367 (17)	171 (4)

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