Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diaquabis(3,7-dichloroquinoline-8-carboxylato)zinc(II) monohydrate

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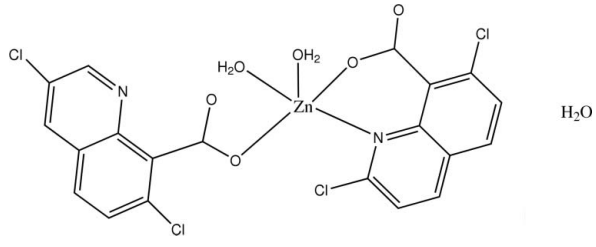
Received 7 August 2008; accepted 8 August 2008

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.041; wR factor = 0.095; data-to-parameter ratio = 14.0.

In the title compound, $[Zn(C_{10}H_4Cl_2NO_2)_2(H_2O)_2]\cdot H_2O$, the Zn atom has a distorted square-pyramidal geometry comprising two O atoms and one N atom from two distinct 3,7-dichloroquinoline-8-carboxylate ligands, and two water molecules. The free water molecules are involved in intermolecular O—H···O hydrogen bonding with the coordinated water molecules and carboxylate O atoms, to give a one-dimensional helical chain along the [100] direction.

Related literature

For related literature, see: Adnan *et al.* (2003); Che *et al.* (2005); Chen *et al.* (2001); Li *et al.* (2008); Lumme *et al.* (1984); Yang *et al.* (2005); Zhang *et al.* (2007).



Experimental

Crystal data

$[Zn(C_{10}H_4Cl_2NO_2)_2(H_2O)_2]\cdot H_2O$
M_r = 601.50
Triclinic, $P\overline{1}$
a = 6.8678 (3) Å
b = 12.6996 (5) Å
c = 12.9317 (5) Å
α = 87.572 (1)°
β = 82.893 (1)°
γ = 82.255 (1)°
V = 1108.63 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.64 mm^{-1}
T = 296 (2) K
0.21 × 0.17 × 0.15 mm

Data collection

Rigaku Mercury diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2001)
T_{min} = 0.725, T_{max} = 0.791
14022 measured reflections
4308 independent reflections
3099 reflections with $I > 2\sigma(I)$
R_{int} = 0.080

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.094
S = 0.97
4308 reflections
307 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5A···O1^{i}	0.85	2.05	2.889 (4)	166
O5—H5B···N2	0.86	2.16	3.002 (4)	169
O6—H6A···O4^{ii}	0.85	1.78	2.628 (4)	174
O6—H6B···O7^{iii}	0.85	1.87	2.677 (4)	156
O7—H7B···O2^{iv}	0.85	2.04	2.827 (4)	153
O7—H7C···O1^{v}	0.85	2.25	3.029 (4)	153

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

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Program for Excellent Talents in Huaiyin Teachers College (grant Nos. ETHYTC and 07QNZC010) and by the Natural Science Foundation of the Education Committee of Guangxi Province.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2255).

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supporting information

Acta Cryst. (2008). E**64**, m1170 [doi:10.1107/S1600536808025671]

Diaquabis(3,7-dichloroquinoline-8-carboxylato)zinc(II) monohydrate

Li-Tao An, Jian Zhou, Jian-Feng Zhou and Min Xia

S1. Comment

Quinolinecarboxylate derivatives and their complexes have attracted considerable interest, because of their interesting high germicidal, antitumoral and pharmacological properties (Adnan *et al.*, 2003; Lumme *et al.*, 1984). Although some transition metal complexes of quinolinecarboxylate ligands have been reported (Che *et al.*, 2005; Chen *et al.*, 2001; Yang *et al.*, 2005), the crystal structures of the 3,7-Dichloro-8-quinolinecarboxylate and its complexes are limited in number, the only two examples are $[M(C_{10}H_4Cl_2NO_2)_2]_n$($M$ = Ni, Co) (Li *et al.*, 2008; Zhang *et al.*, 2007). we report herein on the structure of $[Zn(C_{10}H_4Cl_2NO_2)_2(H_2O)_2].H_2O$, (I).

The title complex, (I) crystallizes in the triclinic space group $P\bar{1}$, with free water molecules in the crystal structure.The Zn(II) center exhibits a distorted square-pyramidal geometry defined by two O atoms and one N atom from two distinct 3,7-Dichloro-8-quinolinecarboxylate ligands, and two water molecules (Fig.1). The N1, O1, O3 and O5 atoms form the base plane, while the O6 atom occupies the axial position.The carboxylate group is bound in a monodentate fashion, with a weak intramolecular O—H···O H-bond between the carboxylate O atom and water molecules. The $[Zn(C_{10}H_4Cl_2NO_2)_2(H_2O)_2]$ molecules are linked *via* these H-bond interactions into a 1-D helical chain along the [100] direction (Fig. 2).

S2. Experimental

The source materials of Zinc hydroxide (0.005 g) and Quinclorac (3,7-Dichloro-8-quinolinecarboxylic acid) (0.024 g) dissolved in 10 ml distilled water and were carefully mixed, and then loaded into a Teflon-lined stainless steel autoclave. The sealed autoclave was heated to 433 K and maintained at this temperature for 48 h. After cooling to room temperature, then some colorless column crystal was obtained.

S3. Refinement

All H atoms were positioned geometrically and were allowed to ride on their parent atoms.

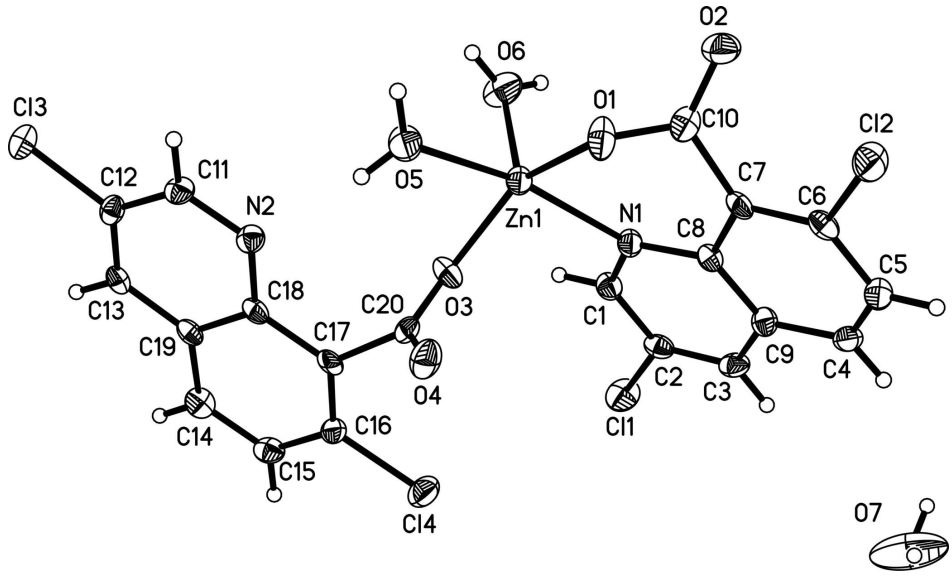

Figure 1

The structure of (I), with the atomic numbering scheme and displacement ellipsoids at the 50% probability level. All H atoms have been omitted for clarity.

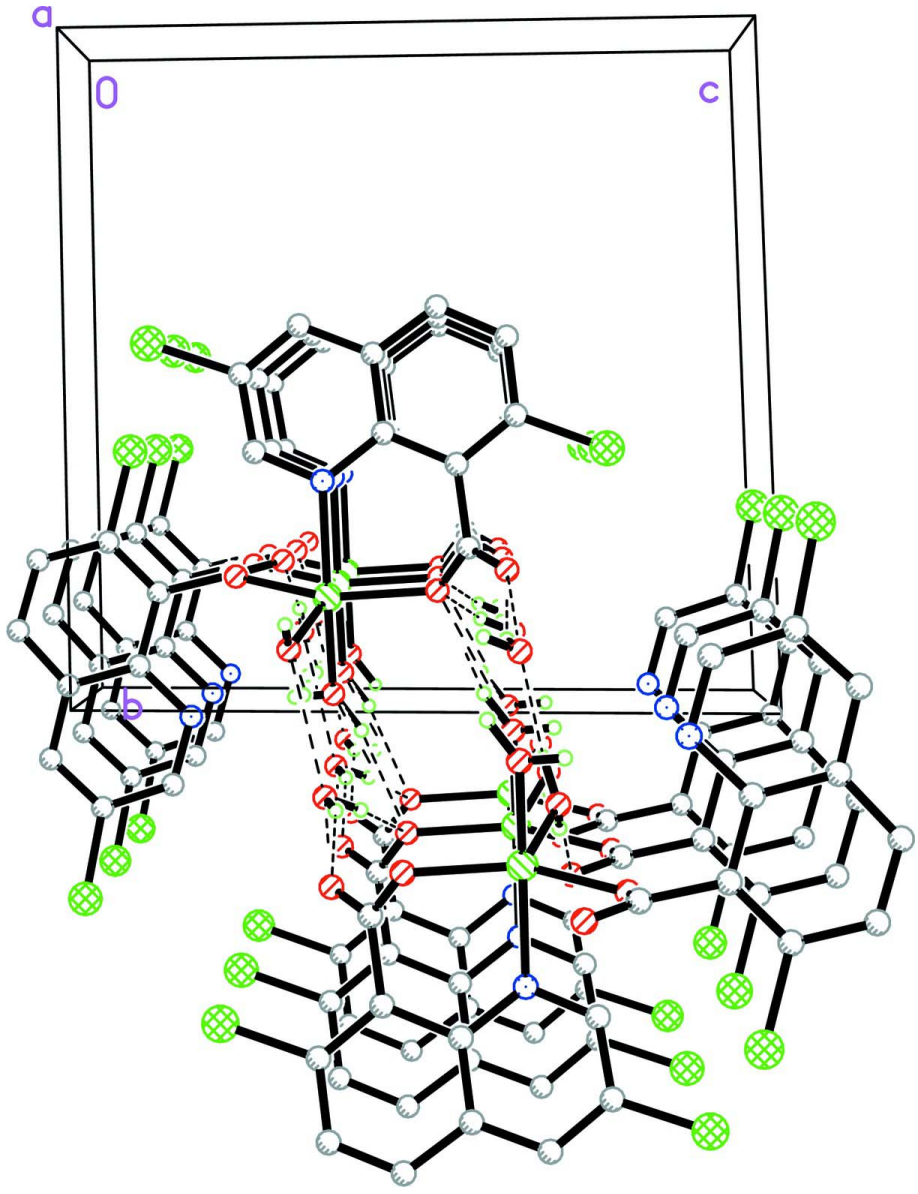

Figure 2

Part of the crystal structure of (I), showing the 1-D helical chain.H atoms bonded to C atoms have been omitted for clarity.

Diaquabis(3,7-dichloroquinoline-8-carboxylato)zinc(II) monohydrate

Crystal data

$[Zn(C_{10}H_4Cl_2NO_2)_2(H_2O)_2]\cdot H_2O$
$M_r = 601.50$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.8678\ (3)$ Å
$b = 12.6996\ (5)$ Å
$c = 12.9317\ (5)$ Å
$\alpha = 87.572\ (1)°$
$\beta = 82.893\ (1)°$
$\gamma = 82.255\ (1)°$
$V = 1108.63\ (8)$ Å^3
$Z = 2$
$F(000) = 604$
$D_x = 1.802$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1973 reflections
$\theta = 1.6–26.0°$
$\mu = 1.64$ mm^{-1}

T = 296 K
Column, colourless

0.21 × 0.17 × 0.15 mm

Data collection

Rigaku Mercury diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2001)
T_{min} = 0.725, T_{max} = 0.791

14022 measured reflections
4308 independent reflections
3099 reflections with $I > 2\sigma(I)$
R_{int} = 0.080
θ_{max} = 26.0°, θ_{min} = 1.6°
h = −8→8
k = −15→15
l = −15→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.094
S = 0.97
4308 reflections
307 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0408P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 e Å^{-3}

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.2965 (5)	0.6256 (3)	0.2794 (3)	0.0197 (8)
H1	0.3070	0.6713	0.2215	0.024*
C2	0.3295 (5)	0.5161 (3)	0.2636 (3)	0.0194 (8)
C3	0.3176 (5)	0.4475 (3)	0.3464 (3)	0.0209 (8)
H3	0.3380	0.3746	0.3366	0.025*
C4	0.2565 (5)	0.4215 (3)	0.5377 (3)	0.0210 (8)
H4	0.2778	0.3480	0.5315	0.025*
C5	0.2088 (5)	0.4641 (3)	0.6336 (3)	0.0241 (9)
H5	0.1921	0.4201	0.6925	0.029*
C6	0.1848 (5)	0.5751 (3)	0.6434 (3)	0.0186 (8)
C7	0.2042 (5)	0.6439 (3)	0.5590 (3)	0.0176 (8)
C8	0.2434 (5)	0.5998 (3)	0.4583 (3)	0.0173 (8)
C9	0.2741 (5)	0.4879 (3)	0.4474 (3)	0.0188 (8)
C10	0.2017 (6)	0.7613 (3)	0.5715 (3)	0.0220 (8)

C11	−0.2472 (5)	1.1168 (3)	0.1346 (3)	0.0194 (8)
H11	−0.2486	1.1675	0.1846	0.023*
C12	−0.2620 (5)	1.1520 (3)	0.0307 (3)	0.0182 (8)
C13	−0.2648 (5)	1.0809 (3)	−0.0443 (3)	0.0179 (8)
H13	−0.2756	1.1034	−0.1129	0.022*
C14	−0.2534 (5)	0.8915 (3)	−0.0880 (3)	0.0221 (8)
H14	−0.2643	0.9098	−0.1577	0.027*
C15	−0.2399 (5)	0.7872 (3)	−0.0559 (3)	0.0213 (8)
H15	−0.2427	0.7346	−0.1034	0.026*
C16	−0.2218 (5)	0.7597 (3)	0.0488 (3)	0.0196 (8)
C17	−0.2162 (5)	0.8341 (3)	0.1213 (3)	0.0167 (8)
C18	−0.2329 (5)	0.9426 (3)	0.0898 (3)	0.0170 (8)
C19	−0.2509 (5)	0.9721 (3)	−0.0157 (3)	0.0172 (8)
C20	−0.1707 (5)	0.8066 (3)	0.2315 (3)	0.0179 (8)
Cl1	0.37704 (15)	0.47230 (7)	0.13739 (7)	0.0300 (2)
Cl2	0.12831 (15)	0.62268 (8)	0.76860 (7)	0.0300 (2)
Cl3	−0.28261 (14)	1.28675 (7)	0.00156 (7)	0.0247 (2)
Cl4	−0.19519 (15)	0.62565 (7)	0.08517 (8)	0.0295 (2)
N1	0.2514 (4)	0.6674 (2)	0.3722 (2)	0.0185 (7)
N2	−0.2316 (4)	1.0165 (2)	0.1644 (2)	0.0190 (7)
O1	0.0705 (4)	0.82485 (19)	0.52664 (19)	0.0294 (7)
O2	0.3222 (4)	0.7909 (2)	0.6231 (2)	0.0325 (7)
O3	0.0098 (3)	0.80624 (19)	0.24133 (19)	0.0222 (6)
O4	−0.3021 (4)	0.78885 (19)	0.3020 (2)	0.0289 (6)
O5	−0.0852 (4)	0.9774 (2)	0.3738 (2)	0.0336 (7)
H5A	−0.0800	1.0295	0.4126	0.040*
H5B	−0.1398	0.9842	0.3175	0.040*
O6	0.3458 (4)	0.9015 (2)	0.3214 (2)	0.0350 (7)
H6A	0.4589	0.8647	0.3195	0.042*
H6B	0.3090	0.9657	0.3391	0.042*
O7	0.3413 (5)	0.1059 (3)	0.3655 (3)	0.0858 (14)
H7B	0.4519	0.1189	0.3815	0.103*
H7C	0.2491	0.1273	0.4129	0.103*
Zn1	0.10699 (6)	0.83389 (3)	0.37255 (3)	0.01835 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.020 (2)	0.0198 (19)	0.019 (2)	0.0004 (15)	−0.0046 (16)	−0.0023 (16)
C2	0.0166 (19)	0.022 (2)	0.020 (2)	−0.0014 (15)	−0.0030 (15)	−0.0076 (15)
C3	0.020 (2)	0.0158 (19)	0.027 (2)	−0.0019 (15)	−0.0032 (16)	−0.0063 (16)
C4	0.019 (2)	0.0162 (18)	0.029 (2)	−0.0035 (15)	−0.0068 (17)	−0.0012 (16)
C5	0.024 (2)	0.023 (2)	0.026 (2)	−0.0050 (16)	−0.0072 (17)	0.0055 (17)
C6	0.0165 (19)	0.026 (2)	0.0133 (19)	−0.0017 (15)	−0.0024 (15)	−0.0039 (15)
C7	0.0130 (18)	0.0215 (19)	0.019 (2)	0.0014 (15)	−0.0071 (15)	−0.0027 (15)
C8	0.0145 (18)	0.0200 (19)	0.017 (2)	−0.0006 (15)	−0.0037 (15)	−0.0004 (15)
C9	0.0177 (19)	0.0184 (19)	0.019 (2)	0.0008 (15)	−0.0025 (15)	−0.0004 (15)
C10	0.032 (2)	0.0191 (19)	0.0123 (19)	0.0004 (17)	0.0038 (17)	−0.0019 (15)

C11	0.020 (2)	0.0167 (19)	0.021 (2)	0.0004 (15)	−0.0006 (16)	−0.0068 (15)
C12	0.0152 (18)	0.0160 (18)	0.022 (2)	0.0006 (15)	0.0008 (15)	0.0017 (15)
C13	0.0128 (18)	0.0232 (19)	0.018 (2)	−0.0009 (15)	−0.0041 (15)	0.0017 (16)
C14	0.023 (2)	0.029 (2)	0.014 (2)	−0.0046 (17)	0.0010 (16)	0.0006 (16)
C15	0.0192 (19)	0.022 (2)	0.023 (2)	−0.0018 (16)	−0.0013 (16)	−0.0082 (16)
C16	0.0161 (19)	0.0153 (18)	0.028 (2)	−0.0011 (15)	−0.0049 (16)	0.0000 (16)
C17	0.0068 (17)	0.0229 (19)	0.021 (2)	−0.0022 (14)	−0.0032 (14)	0.0007 (15)
C18	0.0106 (17)	0.0219 (19)	0.0188 (19)	−0.0006 (14)	−0.0037 (15)	−0.0023 (15)
C19	0.0087 (17)	0.0237 (19)	0.019 (2)	−0.0008 (15)	−0.0023 (14)	−0.0016 (15)
C20	0.022 (2)	0.0102 (17)	0.021 (2)	0.0001 (15)	−0.0038 (16)	−0.0012 (15)
Cl1	0.0431 (6)	0.0251 (5)	0.0207 (5)	−0.0030 (4)	0.0021 (4)	−0.0090 (4)
Cl2	0.0445 (6)	0.0278 (5)	0.0170 (5)	−0.0053 (4)	−0.0006 (4)	−0.0009 (4)
Cl3	0.0300 (5)	0.0170 (5)	0.0251 (5)	0.0009 (4)	−0.0006 (4)	0.0019 (4)
Cl4	0.0426 (6)	0.0176 (5)	0.0297 (6)	−0.0056 (4)	−0.0077 (5)	−0.0008 (4)
N1	0.0214 (16)	0.0166 (15)	0.0173 (17)	0.0003 (13)	−0.0044 (13)	−0.0005 (13)
N2	0.0186 (16)	0.0175 (16)	0.0202 (17)	−0.0007 (13)	−0.0011 (13)	−0.0023 (13)
O1	0.0448 (18)	0.0228 (14)	0.0167 (14)	0.0110 (13)	−0.0039 (13)	−0.0015 (11)
O2	0.0441 (18)	0.0280 (15)	0.0284 (16)	−0.0120 (13)	−0.0078 (14)	−0.0041 (12)
O3	0.0171 (14)	0.0299 (14)	0.0196 (14)	0.0019 (11)	−0.0063 (11)	−0.0047 (11)
O4	0.0284 (15)	0.0272 (15)	0.0289 (16)	−0.0032 (12)	0.0019 (13)	0.0085 (12)
O5	0.0471 (18)	0.0223 (14)	0.0319 (17)	0.0073 (13)	−0.0161 (14)	−0.0108 (12)
O6	0.0202 (15)	0.0234 (15)	0.060 (2)	−0.0003 (12)	−0.0006 (14)	−0.0041 (13)
O7	0.050 (2)	0.075 (3)	0.132 (4)	−0.030 (2)	0.031 (2)	−0.055 (3)
Zn1	0.0206 (2)	0.0172 (2)	0.0168 (2)	−0.00023 (17)	−0.00297 (17)	−0.00061 (17)

Geometric parameters (Å, º)

C1—N1	1.316 (4)	C13—H13	0.9300
C1—C2	1.399 (5)	C14—C15	1.365 (5)
C1—H1	0.9300	C14—C19	1.419 (5)
C2—C3	1.352 (5)	C14—H14	0.9300
C2—Cl1	1.725 (4)	C15—C16	1.400 (5)
C3—C9	1.407 (5)	C15—H15	0.9300
C3—H3	0.9300	C16—C17	1.366 (5)
C4—C5	1.359 (5)	C16—Cl4	1.739 (3)
C4—C9	1.414 (5)	C17—C18	1.414 (5)
C4—H4	0.9300	C17—C20	1.514 (5)
C5—C6	1.406 (5)	C18—N2	1.376 (4)
C5—H5	0.9300	C18—C19	1.415 (5)
C6—C7	1.374 (5)	C20—O4	1.237 (4)
C6—Cl2	1.732 (4)	C20—O3	1.261 (4)
C7—C8	1.421 (5)	N1—Zn1	2.211 (3)
C7—C10	1.504 (5)	O1—Zn1	1.978 (2)
C8—N1	1.377 (4)	O3—Zn1	1.960 (2)
C8—C9	1.419 (5)	O5—Zn1	2.101 (2)
C10—O2	1.231 (4)	O5—H5A	0.85
C10—O1	1.301 (4)	O5—H5B	0.86
C11—N2	1.310 (4)	O6—Zn1	1.982 (2)

C11—C12	1.410 (5)	O6—H6A	0.85
C11—H11	0.9300	O6—H6B	0.85
C12—C13	1.356 (5)	O7—H7B	0.85
C12—Cl3	1.727 (3)	O7—H7C	0.85
C13—C19	1.409 (5)		

N1—C1—C2	123.4 (3)	C14—C15—C16	119.8 (3)
N1—C1—H1	118.3	C14—C15—H15	120.1
C2—C1—H1	118.3	C16—C15—H15	120.1
C3—C2—C1	119.8 (3)	C17—C16—C15	122.2 (3)
C3—C2—Cl1	121.7 (3)	C17—C16—Cl4	119.5 (3)
C1—C2—Cl1	118.5 (3)	C15—C16—Cl4	118.2 (3)
C2—C3—C9	119.2 (3)	C16—C17—C18	118.8 (3)
C2—C3—H3	120.4	C16—C17—C20	123.6 (3)
C9—C3—H3	120.4	C18—C17—C20	117.3 (3)
C5—C4—C9	120.5 (3)	N2—C18—C17	118.0 (3)
C5—C4—H4	119.7	N2—C18—C19	122.2 (3)
C9—C4—H4	119.7	C17—C18—C19	119.9 (3)
C4—C5—C6	119.7 (3)	C13—C19—C18	118.2 (3)
C4—C5—H5	120.1	C13—C19—C14	122.8 (3)
C6—C5—H5	120.1	C18—C19—C14	119.0 (3)
C7—C6—C5	122.6 (3)	O4—C20—O3	125.9 (3)
C7—C6—Cl2	120.7 (3)	O4—C20—C17	121.5 (3)
C5—C6—Cl2	116.7 (3)	O3—C20—C17	112.6 (3)
C6—C7—C8	117.8 (3)	C1—N1—C8	118.3 (3)
C6—C7—C10	121.9 (3)	C1—N1—Zn1	115.1 (2)
C8—C7—C10	120.1 (3)	C8—N1—Zn1	123.8 (2)
N1—C8—C9	121.0 (3)	C11—N2—C18	117.6 (3)
N1—C8—C7	118.9 (3)	C10—O1—Zn1	117.0 (2)
C9—C8—C7	120.1 (3)	C20—O3—Zn1	123.7 (2)
C3—C9—C4	122.6 (3)	Zn1—O5—H5A	124.5
C3—C9—C8	118.3 (3)	Zn1—O5—H5B	109.3
C4—C9—C8	119.1 (3)	H5A—O5—H5B	123.2
O2—C10—O1	124.4 (3)	Zn1—O6—H6A	119.1
O2—C10—C7	118.4 (3)	Zn1—O6—H6B	101.5
O1—C10—C7	117.2 (3)	H6A—O6—H6B	129.9
N2—C11—C12	123.4 (3)	H7B—O7—H7C	110.1
N2—C11—H11	118.3	O3—Zn1—O1	147.92 (11)
C12—C11—H11	118.3	O3—Zn1—O6	101.43 (11)
C13—C12—C11	120.2 (3)	O1—Zn1—O6	110.65 (12)
C13—C12—Cl3	120.9 (3)	O3—Zn1—O5	86.43 (10)
C11—C12—Cl3	118.9 (3)	O1—Zn1—O5	90.82 (10)
C12—C13—C19	118.4 (3)	O6—Zn1—O5	94.10 (11)
C12—C13—H13	120.8	O3—Zn1—N1	87.67 (10)
C19—C13—H13	120.8	O1—Zn1—N1	88.61 (10)
C15—C14—C19	120.3 (3)	O6—Zn1—N1	97.37 (11)
C15—C14—H14	119.8	O5—Zn1—N1	167.94 (11)
C19—C14—H14	119.8		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*A*···O1^{i}	0.85	2.05	2.889 (4)	166
O5—H5*B*···N2	0.86	2.16	3.002 (4)	169
O6—H6*A*···O4^{ii}	0.85	1.78	2.628 (4)	174
O6—H6*B*···O7^{iii}	0.85	1.87	2.677 (4)	156
O7—H7*B*···O2^{iv}	0.85	2.04	2.827 (4)	153
O7—H7*C*···O1^{v}	0.85	2.25	3.029 (4)	153

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