Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

8-Hydroxy-5,7-dimethylquinolin-1-ium chloride dihydrate

Kaliyaperumal Thanigaimani, Nuridayanti Che Khalib, Suhana Arshad and Ibrahim Abdul Razak*‡

School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: arazaki@usm.my

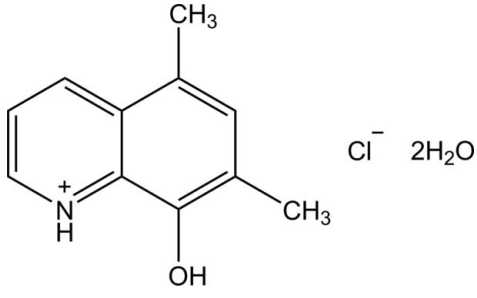
Received 27 November 2012; accepted 3 December 2012

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.001 Å; R factor = 0.028; wR factor = 0.092; data-to-parameter ratio = 19.9.

In the title hydrated salt, $C_{11}H_{12}NO^+\cdot Cl^-\cdot 2H_2O$, the quinoline ring system is essentially planar, with a maximum deviation of 0.005 (1) Å for all non-H atoms. In the crystal, the three components are linked by O—H···O, N—H···O, O—H···Cl and weak C—H···O hydrogen bonds, forming a layer structure parallel to the *ac* plane. The crystal structure is further stabilized by π–π stacking interactions, with centroid–centroid distances of 3.5213 (6) and 3.7176 (6) Å.

Related literature

For background to and the biological activity of quinoline derivatives, see: Balasubramanian & Muthiah (1996*a*,*b*); Morimoto *et al.* (1991); Markees *et al.* (1970). For bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{11}H_{12}NO^+\cdot Cl^-\cdot 2H_2O$
M_r = 245.70
Triclinic, $P\overline{1}$
a = 6.7990 (5) Å
b = 9.2215 (6) Å
c = 10.2123 (7) Å
α = 103.820 (1)°
β = 95.629 (1)°
γ = 105.517 (1)°
V = 590.04 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 100 K
0.38 × 0.20 × 0.14 mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.889, T_{max} = 0.958
9368 measured reflections
3410 independent reflections
3153 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.092
S = 1.08
3410 reflections
171 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1*A*···O2*W*	0.95	2.55	3.2871 (13)	134
O2*W*—H2*W*2···Cl1	0.858 (19)	2.274 (19)	3.1306 (10)	177.4 (18)
O1—H1*O*1···O1*W*	0.874 (19)	1.811 (19)	2.6718 (10)	167.9 (18)
N1—H1*N*1···O1*W*^{i}	0.826 (18)	1.977 (18)	2.7516 (11)	155.9 (17)
O1*W*—H2*W*1···Cl1^{i}	0.83 (2)	2.27 (2)	3.0758 (9)	164.0 (18)
O2*W*—H1*W*2···Cl1^{ii}	0.76 (2)	2.36 (2)	3.1187 (10)	177.7 (18)
O1*W*—H1*W*1···O2*W*^{iii}	0.805 (19)	1.86 (2)	2.6690 (11)	177.2 (19)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors thank the Malaysian Government and Universiti Sains Malaysia (USM) for the research facilities and USM Short Term Grant No. 304/PFIZIK/6312078 to conduct this work. KT thanks The Academy of Sciences for the Developing World and USM for a TWAS–USM fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5226).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Balasubramanian, T. & Muthiah, P. T. (1996*a*). *Acta Cryst.* C**52**, 1017–1019.
Balasubramanian, T. & Muthiah, P. T. (1996*b*). *Acta Cryst.* C**52**, 2072–2073.
Bruker (2009). *SADABS*, *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Markees, D. G., Dewey, V. C. & Kidder, G. W. (1970). *J. Med. Chem.* **13**, 324–326.
Morimoto, Y., Matsuda, F. & Shirahama, H. (1991). *Synlett*, **3**, 202–203.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

‡ Thomson Reuters ResearcherID: A-5599-2009.

supporting information

Acta Cryst. (2013). E**69**, o44 [https://doi.org/10.1107/S1600536812049495]

8-Hydroxy-5,7-dimethylquinolin-1-ium chloride dihydrate

Kaliyaperumal Thanigaimani, Nuridayanti Che Khalib, Suhana Arshad and Ibrahim Abdul Razak

S1. Comment

Recently, much attention has been devoted to the design and synthesis of supramolecular architectures assembled *via* various weak noncovalent interactions in the crystal structures of oxines (8-hydroxyquinoline), their derivatives and their complexes in a variety of crystalline environments (Balasubramanian & Muthiah, 1996*a*,*b*). Oxine is widely used as analytical reagent. Quinolines and their derivatives are very important compounds because of their wide occurrence in natural products (Morimoto *et al.*, 1991) and biologically active compounds (Markees *et al.*, 1970). In order to study potential hydrogen bonding interactions the crystal structure determination of the title compound (I) was carried out.

The asymmetric unit of the title compound, (I) contains a 8-hydroxy-5,7-dimethylquinolin-1-ium cation, a chloride anion and two water molecules as shown in Fig. 1. One proton is transferred from the hydrochloric acid to the atom N1 of 8-hydroxy-5,7-dimethylquinoline during the crystallization, resulting the formation of salt. The quinoline ring system (N1/C1–C9) is planar with a maximum deviation of 0.005 (1) Å at atom C7. The bond lengths (Allen *et al.*, 1987) and angles are normal.

In the crystal packing (Fig. 2), the ion pairs and water molecules are linked *via* O2W—H2W2···Cl1, O1—H1O1···O1W, N1—H1N1···O1W^{i}, O1W—H2W1···Cl1^{i}, O2W—H1W2···Cl1^{ii}, O1W—H1W1···O2W^{iii} and weak C1—H1A···O2W hydrogen bonds (symmetry codes in Table 1), forming a layer. Furthermore, the crystal structure is stabilized by the following π–π interactions: (*a*) between pyridine (N1/C1–C4/C9, centroid *Cg*1) and benzene (C4–C9, centroid *Cg*2) rings *Cg*1···*Cg*2 (1 - *x*, -*y*, 1 - *z*) 3.5213 (6) Å and (*b*) between benzene rings (C4–C9, centroid *Cg*2) *Cg*2···*Cg*2 (-*x*, -*y*, 1 - *z*) 3.7176 (6) Å.

S2. Experimental

To a hot methanol solution (20 ml) of 8-hydroxy-5,7-dimethylquinoline (36 mg, Aldrich) was added a few drops of hydrochloric acid. The solution was warmed over a heating magnetic stirrer hotplate for a few minutes. The resulting solution was allowed to cool slowly at room temperature and crystals of the title compound (I) appeared after a few days.

S3. Refinement

O- and N-bound H atoms were located in a difference Fourier map and were refined freely [O—H = 0.76 (2)–0.873 (19) Å and N—H = 0.828 (19) Å]. The rest of the hydrogen atoms were positioned geometrically (C—H = 0.95–0.98 Å) and were refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). A rotating-group model was used for the methyl group. Eight outliers were omitted (-3 1 0, 4 -6 2, -4 0 5, -4 -2 3, -4 -3 2, -4 1 5, -4 -3 3 and 3 4 1) in the final refinement.

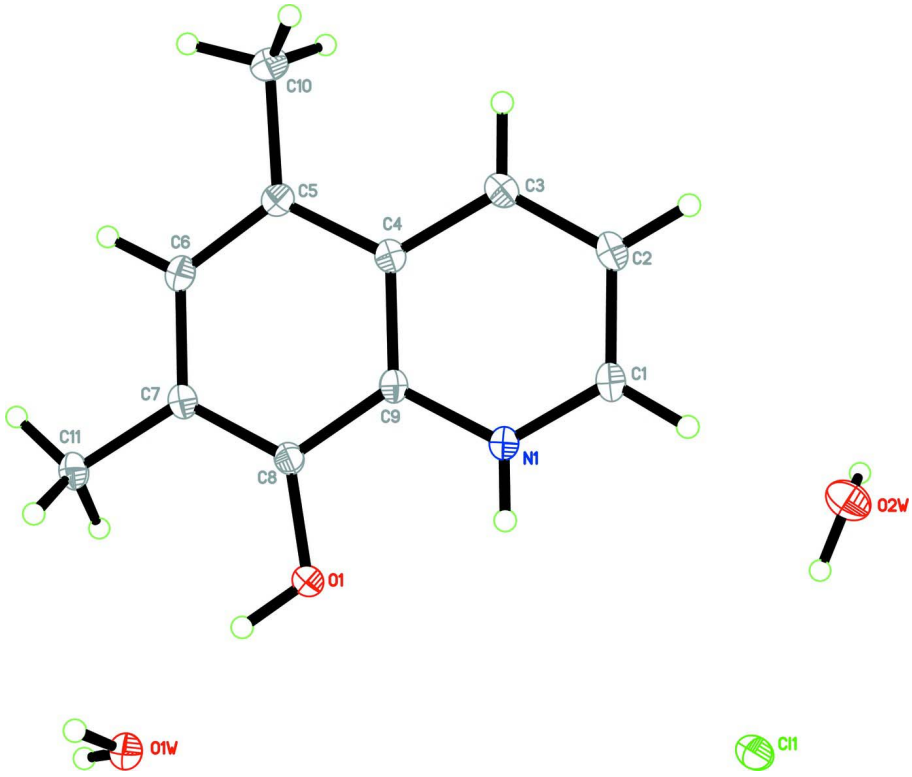


Figure 1

The molecular structure of the title compound with atom labels with 50% probability displacement ellipsoids.

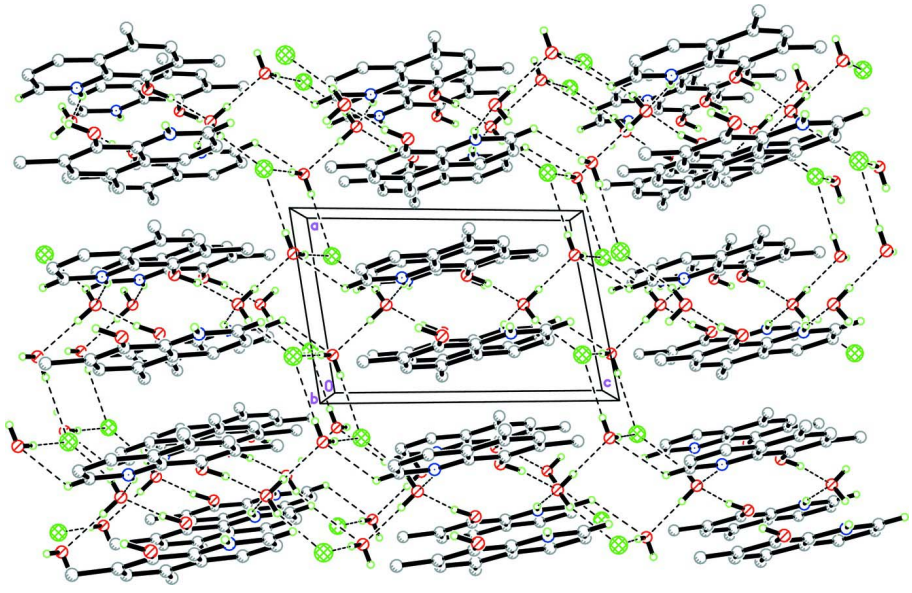


Figure 2

The crystal packing of the title compound. The H atoms not involved in the intermolecular interactions (dashed lines) have been omitted for clarity.

8-Hydroxy-5,7-dimethylquinolin-1-ium chloride dihydrate

Crystal data

$C_{11}H_{12}NO^+·Cl^-·2H_2O$
$M_r = 245.70$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.7990$ (5) Å
$b = 9.2215$ (6) Å
$c = 10.2123$ (7) Å
$\alpha = 103.820$ (1)°
$\beta = 95.629$ (1)°
$\gamma = 105.517$ (1)°
$V = 590.04$ (7) Å^3
$Z = 2$
$F(000) = 260$
$D_x = 1.383$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6250 reflections
$\theta = 2.7$–$30.1°$
$\mu = 0.32$ mm^{-1}
$T = 100$ K
Block, yellow
0.38 × 0.20 × 0.14 mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.889$, $T_{max} = 0.958$
9368 measured reflections
3410 independent reflections
3153 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 30.1°$, $\theta_{min} = 2.1°$
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.092$
$S = 1.08$
3410 reflections
171 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 0.1137P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.22874 (4)	0.70417 (3)	0.91682 (2)	0.01867 (8)

O1W	0.49841 (12)	0.45266 (8)	0.25460 (7)	0.01707 (15)
O1	0.34288 (12)	0.31466 (8)	0.43983 (7)	0.01753 (15)
O2W	0.22470 (13)	0.42047 (10)	1.03561 (8)	0.02223 (16)
N1	0.35726 (12)	0.22535 (9)	0.66748 (8)	0.01260 (15)
C1	0.36928 (14)	0.19382 (11)	0.78767 (9)	0.01493 (17)
H1A	0.4133	0.2770	0.8699	0.018*
C2	0.31746 (15)	0.03900 (11)	0.79353 (9)	0.01541 (17)
H2A	0.3259	0.0159	0.8793	0.018*
C3	0.25385 (14)	−0.08005 (11)	0.67341 (9)	0.01452 (17)
H3A	0.2176	−0.1858	0.6769	0.017*
C4	0.24166 (13)	−0.04734 (10)	0.54510 (9)	0.01229 (16)
C5	0.17880 (14)	−0.16484 (10)	0.41697 (9)	0.01365 (17)
C6	0.17410 (14)	−0.11634 (10)	0.29989 (9)	0.01398 (17)
H6A	0.1328	−0.1942	0.2143	0.017*
C7	0.22731 (13)	0.04326 (10)	0.29896 (9)	0.01296 (17)
C8	0.29038 (13)	0.15788 (10)	0.42249 (9)	0.01246 (16)
C9	0.29632 (13)	0.11195 (10)	0.54492 (9)	0.01168 (16)
C10	0.11810 (16)	−0.33626 (11)	0.41114 (10)	0.01908 (19)
H10A	0.0812	−0.3994	0.3153	0.029*
H10B	−0.0012	−0.3613	0.4573	0.029*
H10C	0.2348	−0.3595	0.4569	0.029*
C11	0.21195 (15)	0.08368 (11)	0.16520 (9)	0.01676 (18)
H11A	0.1307	0.1572	0.1680	0.025*
H11B	0.1439	−0.0118	0.0908	0.025*
H11C	0.3513	0.1320	0.1494	0.025*
H1N1	0.389 (3)	0.318 (2)	0.6664 (18)	0.037 (4)*
H2W1	0.584 (3)	0.411 (2)	0.2240 (19)	0.040 (5)*
H2W2	0.228 (3)	0.497 (2)	1.001 (2)	0.050 (5)*
H1O1	0.385 (3)	0.347 (2)	0.3707 (19)	0.038 (4)*
H1W2	0.115 (3)	0.390 (2)	1.0495 (18)	0.036 (4)*
H1W1	0.416 (3)	0.446 (2)	0.1895 (19)	0.038 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02078 (13)	0.01844 (12)	0.01979 (13)	0.00704 (9)	0.00619 (9)	0.00844 (9)
O1W	0.0219 (3)	0.0155 (3)	0.0134 (3)	0.0053 (3)	0.0038 (3)	0.0035 (2)
O1	0.0275 (4)	0.0127 (3)	0.0126 (3)	0.0050 (3)	0.0048 (3)	0.0046 (2)
O2W	0.0205 (4)	0.0251 (4)	0.0262 (4)	0.0094 (3)	0.0057 (3)	0.0130 (3)
N1	0.0138 (3)	0.0131 (3)	0.0105 (3)	0.0039 (3)	0.0020 (3)	0.0027 (3)
C1	0.0157 (4)	0.0183 (4)	0.0103 (4)	0.0050 (3)	0.0021 (3)	0.0032 (3)
C2	0.0165 (4)	0.0192 (4)	0.0119 (4)	0.0058 (3)	0.0029 (3)	0.0061 (3)
C3	0.0146 (4)	0.0160 (4)	0.0143 (4)	0.0048 (3)	0.0033 (3)	0.0061 (3)
C4	0.0112 (4)	0.0142 (4)	0.0120 (4)	0.0044 (3)	0.0021 (3)	0.0040 (3)
C5	0.0134 (4)	0.0131 (4)	0.0140 (4)	0.0040 (3)	0.0025 (3)	0.0029 (3)
C6	0.0140 (4)	0.0147 (4)	0.0118 (4)	0.0040 (3)	0.0019 (3)	0.0015 (3)
C7	0.0123 (4)	0.0158 (4)	0.0110 (4)	0.0046 (3)	0.0022 (3)	0.0038 (3)
C8	0.0133 (4)	0.0134 (4)	0.0114 (4)	0.0044 (3)	0.0028 (3)	0.0041 (3)

C9	0.0108 (3)	0.0142 (4)	0.0100 (4)	0.0041 (3)	0.0022 (3)	0.0028 (3)
C10	0.0241 (5)	0.0128 (4)	0.0187 (4)	0.0039 (3)	0.0031 (4)	0.0033 (3)
C11	0.0208 (4)	0.0188 (4)	0.0100 (4)	0.0052 (3)	0.0015 (3)	0.0040 (3)

Geometric parameters (Å, º)

O1W—H2W1	0.828 (19)	C4—C9	1.4160 (12)
O1W—H1W1	0.806 (19)	C4—C5	1.4262 (12)
O1—C8	1.3558 (11)	C5—C6	1.3735 (12)
O1—H1O1	0.873 (19)	C5—C10	1.5079 (13)
O2W—H2W2	0.86 (2)	C6—C7	1.4211 (12)
O2W—H1W2	0.76 (2)	C6—H6A	0.9500
N1—C1	1.3271 (11)	C7—C8	1.3809 (12)
N1—C9	1.3686 (11)	C7—C11	1.5006 (12)
N1—H1N1	0.828 (19)	C8—C9	1.4129 (12)
C1—C2	1.3935 (13)	C10—H10A	0.9800
C1—H1A	0.9500	C10—H10B	0.9800
C2—C3	1.3768 (13)	C10—H10C	0.9800
C2—H2A	0.9500	C11—H11A	0.9800
C3—C4	1.4127 (12)	C11—H11B	0.9800
C3—H3A	0.9500	C11—H11C	0.9800
H2W1—O1W—H1W1	106.5 (17)	C7—C6—H6A	118.0
C8—O1—H1O1	116.0 (11)	C8—C7—C6	118.69 (8)
H2W2—O2W—H1W2	107.8 (19)	C8—C7—C11	121.56 (8)
C1—N1—C9	123.25 (8)	C6—C7—C11	119.75 (8)
C1—N1—H1N1	118.5 (12)	O1—C8—C7	126.20 (8)
C9—N1—H1N1	118.2 (12)	O1—C8—C9	115.02 (8)
N1—C1—C2	120.13 (8)	C7—C8—C9	118.75 (8)
N1—C1—H1A	119.9	N1—C9—C8	118.85 (8)
C2—C1—H1A	119.9	N1—C9—C4	118.90 (8)
C3—C2—C1	119.17 (8)	C8—C9—C4	122.25 (8)
C3—C2—H2A	120.4	C5—C10—H10A	109.5
C1—C2—H2A	120.4	C5—C10—H10B	109.5
C2—C3—C4	121.00 (8)	H10A—C10—H10B	109.5
C2—C3—H3A	119.5	C5—C10—H10C	109.5
C4—C3—H3A	119.5	H10A—C10—H10C	109.5
C3—C4—C9	117.54 (8)	H10B—C10—H10C	109.5
C3—C4—C5	123.88 (8)	C7—C11—H11A	109.5
C9—C4—C5	118.58 (8)	C7—C11—H11B	109.5
C6—C5—C4	117.73 (8)	H11A—C11—H11B	109.5
C6—C5—C10	121.45 (8)	C7—C11—H11C	109.5
C4—C5—C10	120.82 (8)	H11A—C11—H11C	109.5
C5—C6—C7	123.99 (8)	H11B—C11—H11C	109.5
C5—C6—H6A	118.0		
C9—N1—C1—C2	−0.37 (14)	C11—C7—C8—O1	0.57 (14)
N1—C1—C2—C3	−0.04 (14)	C6—C7—C8—C9	−1.01 (13)

C1—C2—C3—C4	0.35 (14)	C11—C7—C8—C9	178.35 (8)
C2—C3—C4—C9	−0.27 (13)	C1—N1—C9—C8	−179.47 (8)
C2—C3—C4—C5	179.45 (8)	C1—N1—C9—C4	0.44 (13)
C3—C4—C5—C6	−179.96 (8)	O1—C8—C9—N1	−1.49 (12)
C9—C4—C5—C6	−0.25 (12)	C7—C8—C9—N1	−179.51 (8)
C3—C4—C5—C10	0.53 (13)	O1—C8—C9—C4	178.60 (8)
C9—C4—C5—C10	−179.75 (8)	C7—C8—C9—C4	0.59 (13)
C4—C5—C6—C7	−0.21 (13)	C3—C4—C9—N1	−0.12 (12)
C10—C5—C6—C7	179.30 (8)	C5—C4—C9—N1	−179.85 (8)
C5—C6—C7—C8	0.86 (14)	C3—C4—C9—C8	179.79 (8)
C5—C6—C7—C11	−178.51 (9)	C5—C4—C9—C8	0.06 (13)
C6—C7—C8—O1	−178.78 (8)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O2*W*	0.95	2.55	3.2871 (13)	134
O2*W*—H2*W*2···Cl1	0.858 (19)	2.274 (19)	3.1306 (10)	177.4 (18)
O1—H1*O*1···O1*W*	0.874 (19)	1.811 (19)	2.6718 (10)	167.9 (18)
N1—H1*N*1···O1*W*^{i}	0.826 (18)	1.977 (18)	2.7516 (11)	155.9 (17)
O1*W*—H2*W*1···Cl1^{i}	0.83 (2)	2.27 (2)	3.0758 (9)	164.0 (18)
O2*W*—H1*W*2···Cl1^{ii}	0.76 (2)	2.36 (2)	3.1187 (10)	177.7 (18)
O1*W*—H1*W*1···O2*W*^{iii}	0.805 (19)	1.86 (2)	2.6690 (11)	177.2 (19)

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