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(*E*)-*N*′-(4-Chlorobenzylidene)-1-benzofuran-2-carbohydrazide monohydrate

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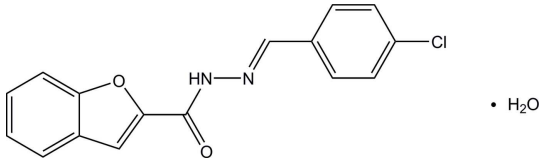
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.073; data-to-parameter ratio = 21.9.

The title compound, $C_{16}H_{11}ClN_2O_2\cdot H_2O$, exists in an *E* conformation with respect to the N=C bond. The benzofuran ring system forms a dihedral angle of 1.26 (4)° with the benzene ring. In the crystal, molecules are linked *via* (N,C)—H···O bifurcated acceptor hydrogen bonds and (O,O,C)—H···O trifurcated acceptor hydrogen bonds, forming layers parallel to the *bc* plane.

Related literature

For general background to hydrazone derivatives, see: Sridhar & Perumal (2003); Vijayakumar *et al.* (2011). For standard bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986). For related structures, see: Fun, Quah & Abdel-Aziz (2012); Fun, Quah, Nitinchandra *et al.* (2012); Fun, Quah, Shyma *et al.* (2012).



Experimental

Crystal data

$C_{16}H_{11}ClN_2O_2\cdot H_2O$
M_r = 316.73
Monoclinic, *Cc*
a = 24.6121 (15) Å
b = 4.6625 (3) Å
c = 12.6570 (8) Å
β = 99.294 (1)°
V = 1433.37 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 100 K
0.57 × 0.34 × 0.09 mm

Data collection

Bruker SMART APEXII DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.856, T_{max} = 0.975
7338 measured reflections
4620 independent reflections
4511 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.073
S = 1.04
4620 reflections
211 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack (1983), 2025 Friedel pairs
Flack parameter: 0.03 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1*W*—H1*W*1···O2	0.855 (19)	2.040 (19)	2.8815 (11)	168.1 (18)
O1*W*—H2*W*1···O2^{i}	0.75 (2)	2.06 (2)	2.8045 (11)	173 (2)
N1—H1*N*1···O1*W*^{ii}	0.909 (19)	1.952 (19)	2.8083 (12)	156.2 (18)
C2—H2*A*···O2^{ii}	0.95	2.57	3.3710 (14)	142
C10—H10*A*···O1*W*^{ii}	0.95	2.54	3.3067 (13)	138

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

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS5157).

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§ Thomson Reuters ResearcherID: A-5525-2009.

supplementary materials

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(*E*)-*N*′-(4-Chlorobenzylidene)-1-benzofuran-2-carbohydrazide monohydrate

Hoong-Kun Fun, Ching Kheng Quah, Nitinchandra, Balakrishna Kalluraya and M. Babu

Comment

Hydrazones are versatile intermediates and important building blocks. Aryl hydrazones are important building blocks for the synthesis of a variety of heterocyclic compounds such as pyrazolines and pyrazoles (Sridhar & Perumal, 2003). Hydrazones of aliphatic and aromatic methyl ketones yield pyrazole-4-carboxaldehyde on formylation by treatment with Vilsmeier reagent. Hydrazones derived from anisaldehyde and 4-nitro-5-ethoxycarbonyl phenylhydrazine showed excellent NLO property (Vijayakumar *et al.*, 2011). Prompted by these observations, the title compound was synthesized and its crystal structure is reported.

The title compound (Fig. 1) consists of a *N*′-[4-chlorophenyl)methylidene]-1-benzofuran-2-carbohydrazide molecule and a water molecule in the asymmetric unit and exists in an *E* configuration with respect to the N2═C10 bond [1.2848 (13) Å]. The benzofuran ring system (O1/C1-C8, r.m.s deviation = 0.012 Å) forms a dihedral angle of 1.26 (4)° with the benzene ring (C11–C16). Bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and are comparable to related structures (Fun, Quah & Abdel-Aziz, 2012; Fun, Quah, Nitinchandra *et al.*, 2012; Fun, Quah, Shyma *et al.*, 2012).

In the crystal (Fig. 2), molecules are linked *via* intermolecular N1—H1N1···O1W, C10—H10A···O1W bifurcated acceptor hydrogen bonds and O1W—H2W1···O2, O1W—H2W1···O2, C2—H2A···O2 trifurcated acceptor hydrogen bonds (Table 1) to form two-dimensional layers parallel to (100).

Experimental

The title compound was obtained by refluxing a mixture of 1-benzofuran-2-carbohydrazide (0.01 mol), 4-chloro-benzaldehyde (0.01 mol) in ethanol (30 ml) and 3 drops of concentrated sulfuric acid for 1 h. Excess ethanol was removed from the reaction mixture under reduced pressure. The solid product obtained was filtered, washed with ethanol and dried. Single crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol-*N*,*N*-dimethyl-formamide (DMF) (3:1) solution.

Refinement

N-bound and O-bound H atoms were located in a difference Fourier map and refined freely [N—H = 0.909 (18) Å, and O—H = 0.75 (3) and 0.857 (19) Å]. The rest of hydrogen atoms were positioned geometrically and refined using a riding model with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C).

Computing details

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

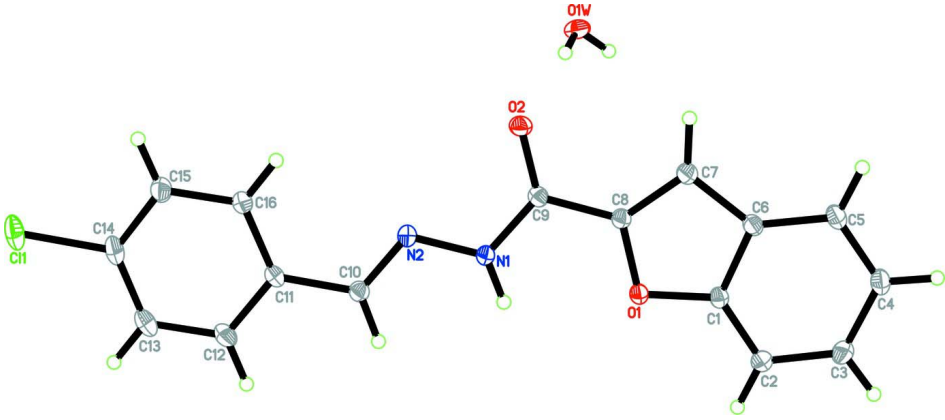

Figure 1

The molecular structure of the title compound showing 50% probability displacement ellipsoids for non-H atoms.

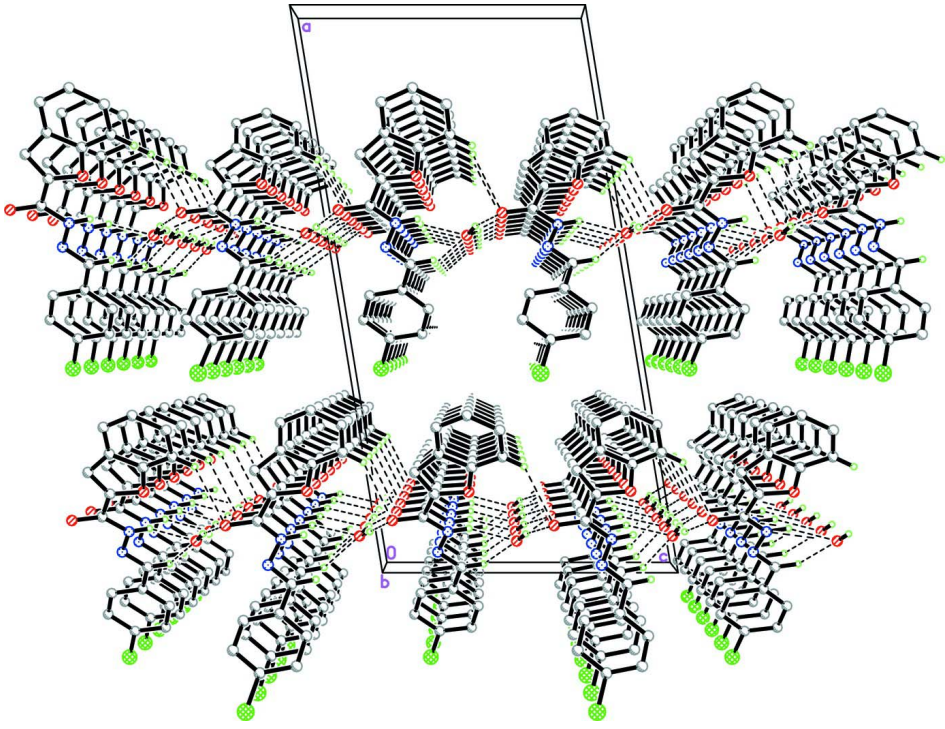

Figure 2

The crystal structure of the title compound, viewed along the *b* axis. H atoms not involved in hydrogen bonds (dashed lines) have been omitted for clarity.

(*E*)-*N*′-(4-Chlorobenzylidene)-1-benzofuran-2-carbohydrazide monohydrate

Crystal data

$C_{16}H_{11}ClN_2O_2·H_2O$
M_r = 316.73
Monoclinic, *Cc*
Hall symbol: C -2yc
a = 24.6121 (15) Å
b = 4.6625 (3) Å
c = 12.6570 (8) Å
β = 99.294 (1)°
V = 1433.37 (16) Å^3
Z = 4
F(000) = 656
D_x = 1.468 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5261 reflections
θ = 3.3–32.6°
μ = 0.28 mm^{-1}
T = 100 K
Plate, yellow
0.57 × 0.34 × 0.09 mm

Data collection

Bruker SMART 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} = 0.856, T_{max} = 0.975
7338 measured reflections
4620 independent reflections
4511 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 32.6°, θ_{min} = 1.7°
h = −35→36
k = −6→7
l = −19→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.073
S = 1.04
4620 reflections
211 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0441P)^2 + 0.2352P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Absolute structure: Flack (1983), 2025 Friedel pairs
Flack parameter: 0.03 (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 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.362378 (12)	2.08150 (5)	0.62418 (2)	0.02405 (7)
O1	0.66851 (3)	0.45040 (16)	0.85030 (6)	0.01364 (14)
O2	0.61809 (3)	0.76090 (16)	0.59506 (6)	0.01549 (14)
N1	0.59805 (4)	0.87204 (18)	0.76135 (7)	0.01207 (15)
N2	0.56127 (4)	1.07881 (18)	0.71696 (7)	0.01267 (15)
C1	0.70999 (4)	0.2512 (2)	0.87293 (8)	0.01263 (16)
C2	0.72801 (5)	0.1294 (2)	0.97208 (8)	0.01605 (18)
H2A	0.7119	0.1758	1.0332	0.019*

C3	0.77114 (5)	−0.0650 (2)	0.97685 (9)	0.0174 (2)
H3A	0.7851	−0.1537	1.0433	0.021*
C4	0.79475 (5)	−0.1341 (2)	0.88576 (9)	0.01846 (19)
H4A	0.8245	−0.2663	0.8922	0.022*
C5	0.77532 (5)	−0.0121 (2)	0.78699 (9)	0.01828 (19)
H5A	0.7911	−0.0607	0.7256	0.022*
C6	0.73181 (4)	0.1849 (2)	0.77992 (8)	0.01348 (17)
C7	0.70075 (4)	0.3521 (2)	0.69570 (8)	0.01474 (17)
H7A	0.7049	0.3542	0.6224	0.018*
C8	0.66431 (4)	0.5062 (2)	0.74233 (8)	0.01243 (16)
C9	0.62453 (4)	0.7225 (2)	0.69371 (8)	0.01223 (17)
C10	0.53404 (4)	1.2045 (2)	0.78210 (8)	0.01311 (17)
H10A	0.5403	1.1551	0.8559	0.016*
C11	0.49318 (4)	1.4248 (2)	0.74216 (8)	0.01304 (17)
C12	0.46196 (5)	1.5490 (2)	0.81314 (9)	0.01721 (19)
H12A	0.4686	1.4950	0.8865	0.021*
C13	0.42134 (5)	1.7509 (2)	0.77787 (10)	0.0192 (2)
H13A	0.3998	1.8321	0.8261	0.023*
C14	0.41298 (4)	1.8308 (2)	0.67104 (10)	0.01754 (19)
C15	0.44432 (5)	1.7160 (2)	0.59957 (9)	0.0181 (2)
H15A	0.4386	1.7771	0.5270	0.022*
C16	0.48413 (4)	1.5106 (2)	0.63496 (8)	0.01574 (18)
H16A	0.5052	1.4284	0.5861	0.019*
O1W	0.58492 (4)	0.25083 (19)	0.47297 (6)	0.01780 (15)
H1W1	0.5989 (8)	0.402 (4)	0.5046 (15)	0.026 (5)*
H2W1	0.5934 (10)	0.127 (5)	0.510 (2)	0.044 (6)*
H1N1	0.6041 (8)	0.820 (4)	0.8315 (15)	0.021 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01451 (11)	0.01539 (10)	0.03983 (16)	0.00472 (8)	−0.00296 (10)	−0.00640 (11)
O1	0.0150 (3)	0.0136 (3)	0.0126 (3)	0.0039 (2)	0.0033 (3)	0.0011 (2)
O2	0.0206 (4)	0.0144 (3)	0.0122 (3)	0.0019 (3)	0.0046 (3)	0.0009 (3)
N1	0.0125 (4)	0.0121 (3)	0.0117 (3)	0.0020 (3)	0.0019 (3)	0.0011 (3)
N2	0.0120 (4)	0.0116 (3)	0.0144 (4)	0.0009 (3)	0.0018 (3)	0.0008 (3)
C1	0.0126 (4)	0.0109 (4)	0.0144 (4)	0.0010 (3)	0.0024 (3)	−0.0005 (3)
C2	0.0175 (5)	0.0169 (4)	0.0137 (4)	0.0026 (4)	0.0024 (4)	0.0009 (3)
C3	0.0179 (5)	0.0170 (4)	0.0164 (5)	0.0023 (3)	0.0004 (4)	0.0019 (3)
C4	0.0165 (5)	0.0183 (4)	0.0207 (5)	0.0060 (4)	0.0034 (4)	0.0012 (4)
C5	0.0186 (5)	0.0187 (4)	0.0185 (5)	0.0062 (4)	0.0060 (4)	−0.0003 (4)
C6	0.0140 (4)	0.0130 (4)	0.0140 (4)	0.0017 (3)	0.0040 (3)	0.0003 (3)
C7	0.0165 (4)	0.0147 (4)	0.0135 (4)	0.0023 (3)	0.0038 (3)	0.0009 (3)
C8	0.0134 (4)	0.0120 (4)	0.0121 (4)	0.0012 (3)	0.0026 (3)	0.0009 (3)
C9	0.0134 (4)	0.0106 (4)	0.0131 (4)	−0.0003 (3)	0.0034 (3)	0.0003 (3)
C10	0.0140 (4)	0.0127 (4)	0.0129 (4)	0.0007 (3)	0.0029 (3)	0.0007 (3)
C11	0.0124 (4)	0.0122 (4)	0.0150 (4)	0.0002 (3)	0.0039 (3)	−0.0022 (3)
C12	0.0193 (5)	0.0159 (4)	0.0180 (4)	0.0011 (4)	0.0077 (4)	−0.0020 (4)
C13	0.0169 (5)	0.0166 (4)	0.0257 (5)	0.0017 (4)	0.0080 (4)	−0.0050 (4)
C14	0.0116 (4)	0.0119 (4)	0.0284 (5)	0.0014 (3)	0.0009 (4)	−0.0040 (4)

C15	0.0175 (5)	0.0170 (4)	0.0187 (5)	0.0039 (3)	0.0000 (4)	−0.0012 (4)
C16	0.0153 (4)	0.0166 (4)	0.0155 (4)	0.0044 (3)	0.0028 (3)	−0.0007 (3)
O1W	0.0256 (4)	0.0159 (3)	0.0116 (3)	−0.0015 (3)	0.0022 (3)	0.0006 (3)

Geometric parameters (Å, º)

Cl1—C14	1.7407 (11)	C6—C7	1.4371 (14)
O1—C1	1.3757 (12)	C7—C8	1.3566 (14)
O1—C8	1.3785 (12)	C7—H7A	0.9500
O2—C9	1.2459 (12)	C8—C9	1.4696 (13)
N1—C9	1.3505 (12)	C10—C11	1.4690 (14)
N1—N2	1.3785 (12)	C10—H10A	0.9500
N1—H1N1	0.909 (18)	C11—C16	1.3975 (14)
N2—C10	1.2848 (13)	C11—C12	1.3984 (14)
C1—C2	1.3837 (14)	C12—C13	1.3931 (16)
C1—C6	1.4049 (13)	C12—H12A	0.9500
C2—C3	1.3896 (15)	C13—C14	1.3856 (18)
C2—H2A	0.9500	C13—H13A	0.9500
C3—C4	1.4097 (16)	C14—C15	1.3876 (16)
C3—H3A	0.9500	C15—C16	1.3916 (15)
C4—C5	1.3863 (16)	C15—H15A	0.9500
C4—H4A	0.9500	C16—H16A	0.9500
C5—C6	1.4025 (14)	O1W—H1W1	0.857 (19)
C5—H5A	0.9500	O1W—H2W1	0.75 (3)
C1—O1—C8	105.55 (8)	C7—C8—C9	128.60 (9)
C9—N1—N2	117.07 (8)	O1—C8—C9	118.87 (8)
C9—N1—H1N1	117.6 (12)	O2—C9—N1	124.39 (9)
N2—N1—H1N1	125.2 (12)	O2—C9—C8	119.11 (9)
C10—N2—N1	115.70 (9)	N1—C9—C8	116.48 (8)
O1—C1—C2	125.76 (9)	N2—C10—C11	119.81 (9)
O1—C1—C6	110.21 (8)	N2—C10—H10A	120.1
C2—C1—C6	124.03 (9)	C11—C10—H10A	120.1
C1—C2—C3	115.97 (10)	C16—C11—C12	119.16 (10)
C1—C2—H2A	122.0	C16—C11—C10	121.81 (9)
C3—C2—H2A	122.0	C12—C11—C10	119.02 (9)
C2—C3—C4	121.76 (10)	C13—C12—C11	120.95 (10)
C2—C3—H3A	119.1	C13—C12—H12A	119.5
C4—C3—H3A	119.1	C11—C12—H12A	119.5
C5—C4—C3	121.07 (10)	C14—C13—C12	118.71 (10)
C5—C4—H4A	119.5	C14—C13—H13A	120.6
C3—C4—H4A	119.5	C12—C13—H13A	120.6
C4—C5—C6	118.35 (10)	C13—C14—C15	121.45 (10)
C4—C5—H5A	120.8	C13—C14—Cl1	119.90 (8)
C6—C5—H5A	120.8	C15—C14—Cl1	118.65 (9)
C5—C6—C1	118.81 (9)	C14—C15—C16	119.50 (10)
C5—C6—C7	135.37 (10)	C14—C15—H15A	120.2
C1—C6—C7	105.81 (9)	C16—C15—H15A	120.2
C8—C7—C6	105.94 (9)	C15—C16—C11	120.20 (10)
C8—C7—H7A	127.0	C15—C16—H16A	119.9

C6—C7—H7A	127.0	C11—C16—H16A	119.9
C7—C8—O1	112.48 (9)	H1W1—O1W—H2W1	107 (2)
C9—N1—N2—C10	175.60 (9)	N2—N1—C9—O2	0.48 (14)
C8—O1—C1—C2	−179.09 (10)	N2—N1—C9—C8	179.00 (8)
C8—O1—C1—C6	0.46 (11)	C7—C8—C9—O2	7.15 (16)
O1—C1—C2—C3	−179.16 (10)	O1—C8—C9—O2	−175.52 (9)
C6—C1—C2—C3	1.35 (16)	C7—C8—C9—N1	−171.45 (10)
C1—C2—C3—C4	−0.30 (16)	O1—C8—C9—N1	5.88 (13)
C2—C3—C4—C5	−0.69 (18)	N1—N2—C10—C11	−179.00 (8)
C3—C4—C5—C6	0.67 (17)	N2—C10—C11—C16	−2.28 (15)
C4—C5—C6—C1	0.32 (16)	N2—C10—C11—C12	176.82 (10)
C4—C5—C6—C7	−179.70 (12)	C16—C11—C12—C13	1.54 (16)
O1—C1—C6—C5	179.05 (9)	C10—C11—C12—C13	−177.59 (10)
C2—C1—C6—C5	−1.38 (16)	C11—C12—C13—C14	−1.15 (16)
O1—C1—C6—C7	−0.94 (11)	C12—C13—C14—C15	−0.46 (17)
C2—C1—C6—C7	178.62 (10)	C12—C13—C14—Cl1	179.76 (8)
C5—C6—C7—C8	−178.95 (12)	C13—C14—C15—C16	1.64 (17)
C1—C6—C7—C8	1.04 (11)	Cl1—C14—C15—C16	−178.57 (9)
C6—C7—C8—O1	−0.81 (12)	C14—C15—C16—C11	−1.23 (16)
C6—C7—C8—C9	176.66 (10)	C12—C11—C16—C15	−0.34 (16)
C1—O1—C8—C7	0.24 (11)	C10—C11—C16—C15	178.77 (10)
C1—O1—C8—C9	−177.51 (9)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*1···O2	0.855 (19)	2.040 (19)	2.8815 (11)	168.1 (18)
O1*W*—H2*W*1···O2^{i}	0.75 (2)	2.06 (2)	2.8045 (11)	173 (2)
N1—H1*N*1···O1*W*^{ii}	0.909 (19)	1.952 (19)	2.8083 (12)	156.2 (18)
C2—H2*A*···O2^{ii}	0.95	2.57	3.3710 (14)	142
C10—H10*A*···O1*W*^{ii}	0.95	2.54	3.3067 (13)	138

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