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N′-[(*E*)-1-(3,5-Dichloro-2-hydroxyphenyl)ethylidene]-4-methoxybenzohydrazide monohydrate

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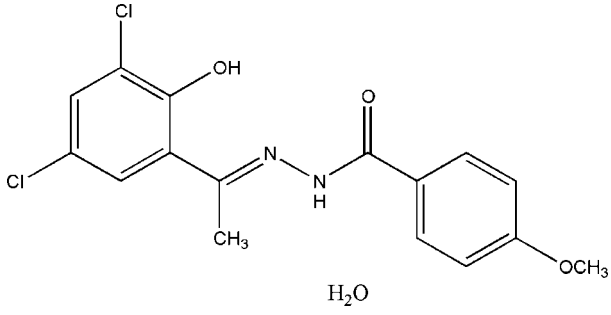
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.066; wR factor = 0.170; data-to-parameter ratio = 13.3.

The title compound, $C_{16}H_{14}Cl_2N_2O_3\cdot H_2O$, displays a *trans* conformation with respect to the C=N double bond. The dihedral angle between the two benzene rings is 4.98 (12)°. Intramolecular O—H···N and O—H···O hydrogen bonds occur. The crystal structure is stabilized by intermolecular O—H···O and N—H···O hydrogen bonds. In addition, there are π–π interactions between the chemically distinct benzene rings of inversion-related molecules [centroid–centroid separation = 3.715 (1) Å].

Related literature

For further details of the chemistry of the title compound, see: Carcelli *et al.* (1995); Salem (1998). For a related stucture, see: Chang *et al.* (2007).



Experimental

Crystal data

$C_{16}H_{14}Cl_2N_2O_3\cdot H_2O$
M_r = 371.21
Triclinic, $P\overline{1}$
a = 7.033 (5) Å
b = 7.516 (7) Å
c = 16.647 (10) Å
α = 85.105 (10)°
β = 81.386 (12)°
γ = 79.414 (10)°
V = 853.7 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 298 K
0.30 × 0.23 × 0.16 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.906, T_{max} = 0.946
4423 measured reflections
2936 independent reflections
1997 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.170
S = 1.00
2936 reflections
220 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O4	0.86	2.15	2.926 (5)	150
O1—H1···O2	0.82	2.58	3.287 (5)	146
O1—H1···N1	0.82	1.77	2.484 (5)	145
O4—H16···O1^{i}	0.85	2.09	2.887 (5)	156
O4—H15···O2^{ii}	0.85	1.88	2.726 (5)	176

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2270).

References

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supporting information

Acta Cryst. (2010). E**66**, o2667 [doi:10.1107/S1600536810038328]

N′-[(*E*)-1-(3,5-Dichloro-2-hydroxyphenyl)ethylidene]-4-methoxybenzohydrazide monohydrate

Chun-Hong He, Jian-Ping Zhang and Jian-Guo Chang

S1. Comment

The chemistry of aroylhydrazones continues to attract much attention due to their coordination ability to metal ions and their biological activity (Carcelli *et al.*, 1995; Salem, 1998; Chang *et al.*, 2007). As an extension of work on the structural characterization of aroylhydrazone derivatives, the title compound, was synthesized and its crystal structure is reported here.

The title molecule displays a *trans* conformation with respect to the C7=N1 double bond (Fig. 1). The dihedral angle between the two benzene rings is 4.98 (12) °. The crystal structure is stabilized by intramolecular O—H···N, O—H···O and intermolecular O—H···O, N—H···O hydrogen bonds. (Table. 1, Figs. 1 and 2). There are π - π interactions between the chemically distinct benzene rings on inversion related molecules [*Cg*···*Cg* = 3.715 (1) Å; *Cg* represents a ring centroid].

S2. Experimental

4-methoxybenzohydrazide (0.01 mol,1.66 g) was dissolved in anhydrous ethanol (50 ml), and 1-(3,5-dichloro-2-hydroxyphenyl)ethanone (0.01 mol, 2.05 g) was added. The reaction mixture was refluxed for 5 h with stirring, then the resulting precipitate was collected by filtration, washed several times with ethanol and dried *in vacuo* (yield 78%). The compound (1.0 mmol,0.35 g) was dissolved in dimethylformamide (30 ml) and kept at room temperature for 20d to obtain yellow single crystals suitable for X-ray diffraction.

S3. Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H (methyl) = 0.96 Å, C—H (aromatic) = 0.93 Å, O—H = 0.82 Å, N—H = 0.86 Å and with U_{iso}(H) = 1.5U_{eq}(C_{methyl}, O and 1.2U_{eq}($C_{aromatic}$, $C_{methylene}$, N).

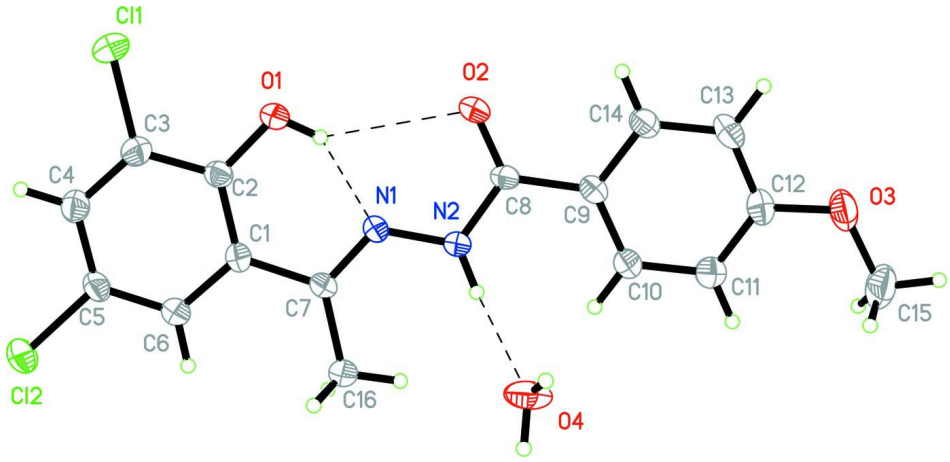

Figure 1

The molecular structure of compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30° probability level. Dashed lines show intramolecular O—H···N, O—H···O and intermolecular N—H···O hydrogen bonds.

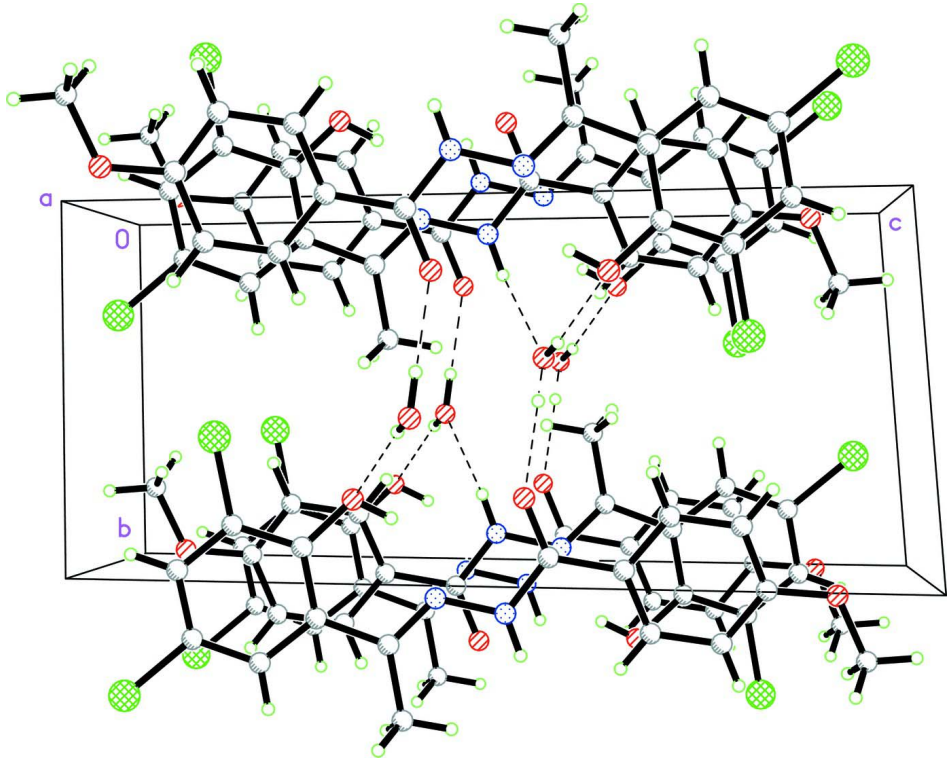

Figure 2

Packing diagram of compound, Showing intermolecular O—H···O and N—H···O hydrogen bonds (dashed lines).

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Triclinic, $P\overline{1}$
Hall symbol: -P 1

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b = 7.516 (7) Å
c = 16.647 (10) Å
α = 85.105 (10)°
β = 81.386 (12)°
γ = 79.414 (10)°
V = 853.7 (11) Å^3
Z = 2
F(000) = 384

D_x = 1.444 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1429 reflections
θ = 3.0–25.5°
μ = 0.40 mm^{-1}
T = 298 K
Plate, yellow
0.30 × 0.23 × 0.16 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.906, T_{max} = 0.946

4423 measured reflections
2936 independent reflections
1997 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.1°, θ_{min} = 2.5°
h = −8→8
k = −6→8
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.170
S = 1.00
2936 reflections
220 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
$w = 1/[\sigma^2(F_o^2) + (0.0727P)^2 + 0.8688P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 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}
Cl1	1.0655 (2)	−0.37144 (18)	0.19059 (9)	0.0675 (5)
Cl2	0.8926 (3)	0.3075 (2)	0.05875 (8)	0.0774 (6)
O1	0.9149 (5)	−0.2090 (4)	0.34325 (19)	0.0462 (8)
H1	0.8675	−0.1599	0.3856	0.069*
O2	0.7613 (6)	−0.2102 (4)	0.5397 (2)	0.0611 (10)
O3	0.5111 (6)	0.0452 (6)	0.8985 (2)	0.0714 (12)
O4	0.7853 (6)	0.4252 (4)	0.5716 (3)	0.0710 (12)
N1	0.7805 (5)	0.0497 (5)	0.4303 (2)	0.0367 (9)

N2	0.7259 (5)	0.0879 (5)	0.5112 (2)	0.0392 (9)
H2	0.6983	0.1973	0.5264	0.047*
C1	0.8427 (6)	0.1022 (6)	0.2902 (3)	0.0360 (9)
C2	0.9122 (6)	−0.0844 (6)	0.2809 (3)	0.0374 (10)
C3	0.9789 (7)	−0.1428 (6)	0.2023 (3)	0.0442 (11)
C4	0.9757 (7)	−0.0251 (7)	0.1341 (3)	0.0488 (11)
H4	1.0221	−0.0668	0.0826	0.059*
C5	0.9020 (7)	0.1566 (7)	0.1441 (3)	0.0448 (11)
C6	0.8384 (7)	0.2200 (6)	0.2203 (3)	0.0419 (10)
H6	0.7918	0.3432	0.2255	0.050*
C7	0.7781 (6)	0.1720 (6)	0.3720 (3)	0.0357 (9)
C8	0.7186 (6)	−0.0562 (6)	0.5645 (3)	0.0387 (10)
C9	0.6577 (6)	−0.0211 (6)	0.6511 (3)	0.0387 (10)
C10	0.5662 (7)	0.1480 (7)	0.6775 (3)	0.0452 (11)
H10	0.5393	0.2442	0.6396	0.054*
C11	0.5141 (7)	0.1750 (7)	0.7606 (3)	0.0508 (12)
H11	0.4542	0.2887	0.7780	0.061*
C12	0.5526 (7)	0.0310 (7)	0.8165 (3)	0.0483 (11)
C13	0.6383 (7)	−0.1378 (7)	0.7914 (3)	0.0517 (12)
H13	0.6614	−0.2346	0.8294	0.062*
C14	0.6904 (7)	−0.1634 (7)	0.7088 (3)	0.0457 (11)
H14	0.7484	−0.2780	0.6919	0.055*
C15	0.4310 (10)	0.2206 (10)	0.9292 (4)	0.0819 (19)
H15A	0.5202	0.3029	0.9109	0.123*
H15B	0.4107	0.2103	0.9876	0.123*
H15C	0.3086	0.2657	0.9095	0.123*
C16	0.7148 (6)	0.3729 (4)	0.3805 (3)	0.0605 (15)
H16A	0.6850	0.3969	0.4371	0.091*
H16B	0.6008	0.4148	0.3542	0.091*
H16C	0.8183	0.4351	0.3555	0.091*
H15	0.7839	0.5382	0.5611	0.14 (3)*
H16	0.8905	0.3884	0.5919	0.09 (2)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0958 (12)	0.0380 (7)	0.0647 (9)	−0.0048 (7)	−0.0002 (8)	−0.0142 (6)
Cl2	0.1369 (15)	0.0568 (9)	0.0331 (7)	−0.0125 (9)	−0.0067 (8)	0.0091 (6)
O1	0.067 (2)	0.0312 (17)	0.0383 (18)	−0.0054 (15)	−0.0049 (16)	0.0012 (14)
O2	0.104 (3)	0.0296 (18)	0.046 (2)	−0.0090 (18)	−0.0052 (19)	0.0065 (15)
O3	0.089 (3)	0.084 (3)	0.034 (2)	−0.001 (2)	−0.0038 (19)	0.0013 (19)
O4	0.090 (3)	0.037 (2)	0.096 (3)	−0.0169 (19)	−0.044 (3)	0.007 (2)
N1	0.050 (2)	0.031 (2)	0.0279 (19)	−0.0080 (16)	−0.0040 (16)	0.0009 (15)
N2	0.053 (2)	0.0283 (19)	0.035 (2)	−0.0060 (16)	−0.0037 (17)	−0.0006 (16)
C1	0.038 (2)	0.039 (2)	0.032 (2)	−0.0095 (18)	−0.0057 (17)	−0.0004 (17)
C2	0.041 (2)	0.033 (2)	0.038 (2)	−0.0108 (18)	−0.0052 (18)	0.0006 (18)
C3	0.048 (2)	0.041 (2)	0.045 (2)	−0.0085 (19)	−0.005 (2)	−0.007 (2)
C4	0.057 (3)	0.049 (3)	0.040 (2)	−0.013 (2)	−0.003 (2)	−0.006 (2)

C5	0.058 (3)	0.043 (2)	0.034 (2)	−0.013 (2)	−0.007 (2)	0.0028 (19)
C6	0.048 (2)	0.038 (2)	0.038 (2)	−0.0076 (19)	−0.0049 (19)	0.0003 (19)
C7	0.043 (2)	0.031 (2)	0.033 (2)	−0.0059 (17)	−0.0058 (18)	0.0025 (17)
C8	0.047 (2)	0.029 (2)	0.039 (2)	−0.0067 (18)	−0.0073 (19)	0.0035 (18)
C9	0.040 (2)	0.040 (2)	0.037 (2)	−0.0101 (18)	−0.0062 (18)	0.0037 (18)
C10	0.051 (3)	0.043 (2)	0.039 (2)	−0.005 (2)	−0.005 (2)	0.002 (2)
C11	0.057 (3)	0.047 (3)	0.046 (3)	−0.006 (2)	−0.002 (2)	−0.002 (2)
C12	0.048 (3)	0.059 (3)	0.037 (2)	−0.008 (2)	−0.005 (2)	0.001 (2)
C13	0.051 (3)	0.058 (3)	0.043 (2)	−0.006 (2)	−0.008 (2)	0.011 (2)
C14	0.050 (3)	0.044 (2)	0.041 (2)	−0.006 (2)	−0.005 (2)	0.006 (2)
C15	0.100 (5)	0.092 (5)	0.048 (3)	−0.004 (4)	0.003 (3)	−0.018 (3)
C16	0.098 (4)	0.035 (3)	0.041 (3)	−0.001 (3)	−0.001 (3)	−0.001 (2)

Geometric parameters (Å, º)

Cl1—C3	1.731 (5)	C5—C6	1.376 (7)
Cl2—C5	1.742 (5)	C6—H6	0.9300
O1—C2	1.338 (5)	C7—C16	1.506 (5)
O1—H1	0.8200	C8—C9	1.474 (6)
O2—C8	1.231 (5)	C9—C14	1.385 (6)
O3—C12	1.362 (6)	C9—C10	1.391 (6)
O3—C15	1.440 (7)	C10—C11	1.399 (7)
O4—H15	0.8511	C10—H10	0.9300
O4—H16	0.8496	C11—C12	1.381 (7)
N1—C7	1.279 (5)	C11—H11	0.9300
N1—N2	1.383 (5)	C12—C13	1.372 (7)
N2—C8	1.344 (5)	C13—C14	1.389 (7)
N2—H2	0.8600	C13—H13	0.9300
C1—C6	1.401 (6)	C14—H14	0.9300
C1—C2	1.412 (6)	C15—H15A	0.9600
C1—C7	1.477 (6)	C15—H15B	0.9600
C2—C3	1.401 (6)	C15—H15C	0.9600
C3—C4	1.378 (7)	C16—H16A	0.9600
C4—C5	1.383 (7)	C16—H16B	0.9600
C4—H4	0.9300	C16—H16C	0.9600
C2—O1—H1	109.5	C14—C9—C10	118.5 (4)
C12—O3—C15	118.7 (5)	C14—C9—C8	118.6 (4)
H15—O4—H16	104.1	C10—C9—C8	122.9 (4)
C7—N1—N2	123.2 (4)	C9—C10—C11	120.7 (5)
C8—N2—N1	115.9 (4)	C9—C10—H10	119.7
C8—N2—H2	122.0	C11—C10—H10	119.7
N1—N2—H2	122.0	C12—C11—C10	119.3 (5)
C6—C1—C2	118.5 (4)	C12—C11—H11	120.4
C6—C1—C7	120.8 (4)	C10—C11—H11	120.4
C2—C1—C7	120.7 (4)	O3—C12—C13	115.8 (5)
O1—C2—C3	118.2 (4)	O3—C12—C11	123.4 (5)
O1—C2—C1	123.3 (4)	C13—C12—C11	120.8 (5)

C3—C2—C1	118.5 (4)	C12—C13—C14	119.6 (5)
C4—C3—C2	122.3 (4)	C12—C13—H13	120.2
C4—C3—Cl1	119.1 (4)	C14—C13—H13	120.2
C2—C3—Cl1	118.6 (4)	C9—C14—C13	121.2 (5)
C3—C4—C5	118.5 (4)	C9—C14—H14	119.4
C3—C4—H4	120.8	C13—C14—H14	119.4
C5—C4—H4	120.8	O3—C15—H15A	109.5
C6—C5—C4	121.1 (4)	O3—C15—H15B	109.5
C6—C5—Cl2	119.6 (4)	H15A—C15—H15B	109.5
C4—C5—Cl2	119.3 (4)	O3—C15—H15C	109.5
C5—C6—C1	121.1 (4)	H15A—C15—H15C	109.5
C5—C6—H6	119.5	H15B—C15—H15C	109.5
C1—C6—H6	119.5	C7—C16—H16A	109.5
N1—C7—C1	114.5 (4)	C7—C16—H16B	109.5
N1—C7—C16	125.9 (4)	H16A—C16—H16B	109.5
C1—C7—C16	119.6 (4)	C7—C16—H16C	109.5
O2—C8—N2	119.6 (4)	H16A—C16—H16C	109.5
O2—C8—C9	122.8 (4)	H16B—C16—H16C	109.5
N2—C8—C9	117.6 (4)		

C7—N1—N2—C8	173.8 (4)	C6—C1—C7—C16	−2.6 (6)
C6—C1—C2—O1	−177.2 (4)	C2—C1—C7—C16	176.7 (4)
C7—C1—C2—O1	3.5 (6)	N1—N2—C8—O2	0.7 (6)
C6—C1—C2—C3	1.7 (6)	N1—N2—C8—C9	−179.0 (4)
C7—C1—C2—C3	−177.5 (4)	O2—C8—C9—C14	14.7 (7)
O1—C2—C3—C4	177.9 (4)	N2—C8—C9—C14	−165.6 (4)
C1—C2—C3—C4	−1.1 (7)	O2—C8—C9—C10	−164.4 (5)
O1—C2—C3—Cl1	−1.4 (6)	N2—C8—C9—C10	15.3 (6)
C1—C2—C3—Cl1	179.7 (3)	C14—C9—C10—C11	2.0 (7)
C2—C3—C4—C5	−0.7 (7)	C8—C9—C10—C11	−178.9 (4)
Cl1—C3—C4—C5	178.5 (4)	C9—C10—C11—C12	−0.7 (7)
C3—C4—C5—C6	1.9 (7)	C15—O3—C12—C13	176.6 (5)
C3—C4—C5—Cl2	−178.9 (4)	C15—O3—C12—C11	−2.9 (8)
C4—C5—C6—C1	−1.3 (7)	C10—C11—C12—O3	178.5 (5)
Cl2—C5—C6—C1	179.6 (4)	C10—C11—C12—C13	−1.0 (8)
C2—C1—C6—C5	−0.6 (7)	O3—C12—C13—C14	−178.2 (4)
C7—C1—C6—C5	178.7 (4)	C11—C12—C13—C14	1.3 (8)
N2—N1—C7—C1	179.8 (4)	C10—C9—C14—C13	−1.7 (7)
N2—N1—C7—C16	−0.8 (7)	C8—C9—C14—C13	179.2 (4)
C6—C1—C7—N1	176.9 (4)	C12—C13—C14—C9	0.1 (7)
C2—C1—C7—N1	−3.9 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O4	0.86	2.15	2.926 (5)	150
O1—H1···O2	0.82	2.58	3.287 (5)	146
O1—H1···N1	0.82	1.77	2.484 (5)	145

O4—H16···O1^{i}	0.85	2.09	2.887 (5)	156
O4—H15···O2^{ii}	0.85	1.88	2.726 (5)	176

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