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

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

Department of Material Science and Chemical Engineering, Taishan University, 271021 Taian, Shandong, People's Republic of China
Correspondence e-mail: tsucjg@163.com

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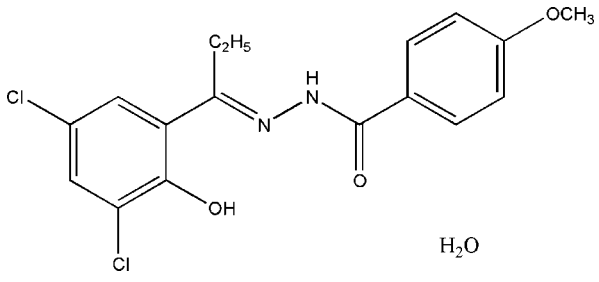
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.042; wR factor = 0.127; data-to-parameter ratio = 14.1.

The title compound, $C_{17}H_{16}Cl_2N_2O_3\cdot H_2O$, displays a *trans* conformation with respect to the C=N double bond. The dihedral angle between the two benzene rings is 30.77 (5)° and there is one intramolecular N—H···O hydrogen bond. In the crystal, molecules are linked by hydrogen bonding to the water molecules of crystallization, which acts as both an acceptor and a donor, into a three-dimensional network.

Related literature

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



Experimental

Crystal data

$C_{17}H_{16}Cl_2N_2O_3\cdot H_2O$ M_r = 385.23
Monoclinic, $C2/c$
a = 32.925 (3) Å
b = 7.3733 (7) Å
c = 15.1252 (13) Å
β = 92.319 (2)°
V = 3668.9 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 295 K
0.21 × 0.16 × 0.11 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.925, T_{max} = 0.960
9328 measured reflections
3242 independent reflections
2320 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.127
S = 1.01
3242 reflections
230 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.77	2.494 (2)	146
N2—H2···O4^{i}	0.86	2.16	3.004 (2)	168
O4—H18···O2^{ii}	0.85	1.97	2.815 (2)	171
O4—H19···O1^{iii}	0.85	2.11	2.908 (2)	155

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

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: FL2314).

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supporting information

Acta Cryst. (2010). E**66**, o2620 [doi:10.1107/S1600536810037293]

N′-[(1*E*)-1-(3,5-Dichloro-2-hydroxyphenyl)propylidene]-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 ability to coordinate with metal ions (Salem, 1998) and their biological activity (Carcelli *et al.*, 1995). As an extension of work on the structural characterization of aroylhydrazone derivatives (Chang & Ji, 2007), the title compound, (I),was synthesized and its crystal structure is reported here.

(I) displays a trans conformation with respect to the C7=N1 double bond (Fig. 1). The N2-N1=C7-C6 torsion angle is -175.44 (18)°.The dihedral angle between the two benzene rings is 30.77 (molecules are linked into a three dimensional network by intermolecular N-H···O, O-H···O Hydrogen bonds involving the water molecule which acts as both a donor and an acceptor.(Table. 1, Fig. 1and Fig. 2).

S2. Experimental

4-methoxybenzohydrazide (0.01 mol,1.66 g) was dissolved in anhydrous ethanol (40 ml), and 1-(3,5-dichloro-2-hydroxyphenyl)propan-1-one (0.01 mol, 2.19 g) was added. The reaction mixture was refluxed for 6 h with stirring, then the resulting precipitate was collected by filtration, washed several times with ethanol and dried *in vacuo* (yield 82%). The compound (1.0 mmol, 0.37 g) was dissolved in dimethylformamide (15 ml) and kept at room temperature for 30 d to obtain colourless 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*(methylene) = 0.97 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*).

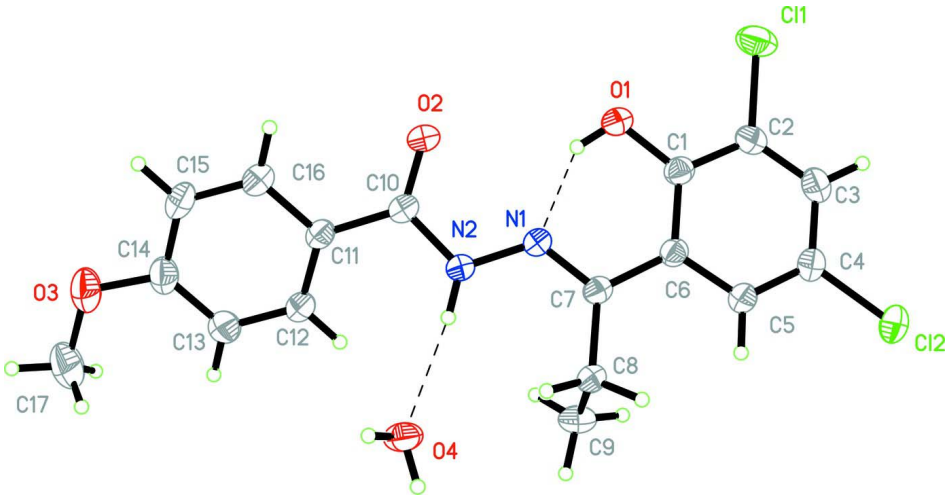

Figure 1

The molecular structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.Dashed lines show intramolecular O–H···N hydrogen bonds.

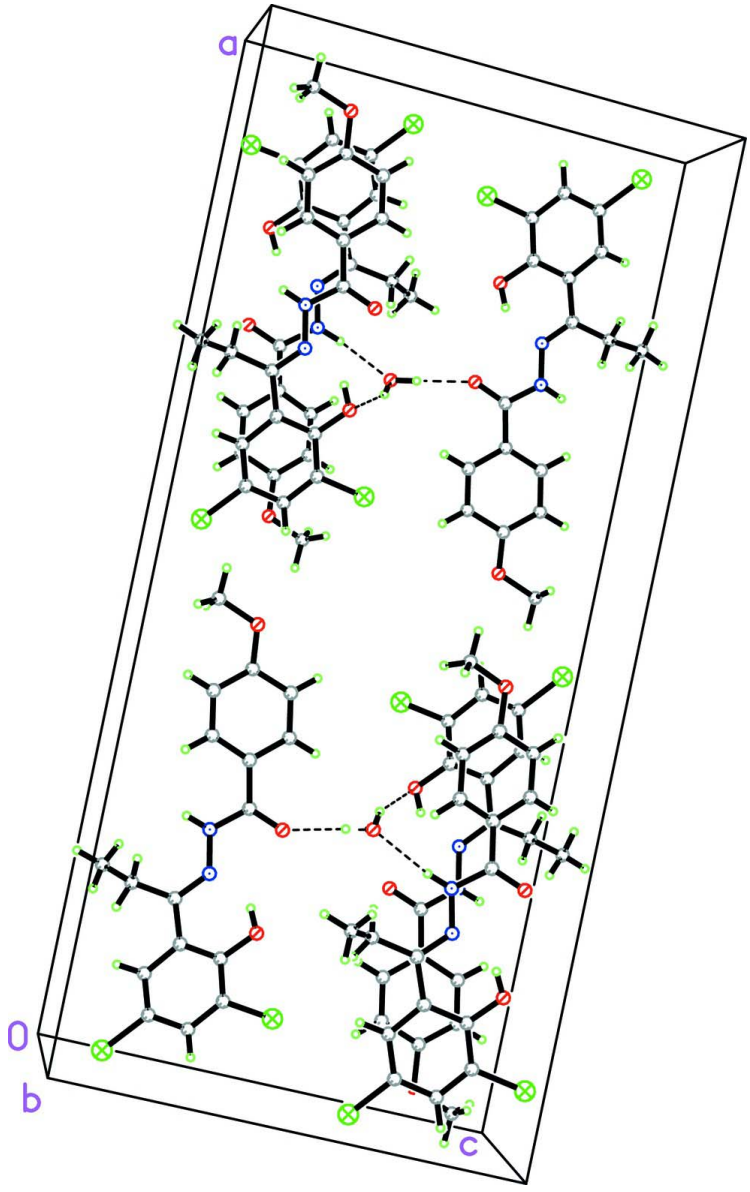

Figure 2

Packing diagram of (I), showing intermolecular O—H···O and N—H···O hydrogen bonds with the water molecule (dashed lines).

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β = 92.319 (2)°
V = 3668.9 (6) Å^3
Z = 8
F(000) = 1600
D_x = 1.395 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 2261 reflections
θ = 2.7–22.8°
μ = 0.38 mm^{-1}

T = 295 K
Block, colourless

0.21 × 0.16 × 0.11 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} = 0.925, T_{max} = 0.960

9328 measured reflections
3242 independent reflections
2320 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.1°, θ_{min} = 2.5°
h = −38→31
k = −7→8
l = −17→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.127
S = 1.01
3242 reflections
230 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
$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 2.1699P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008)
Extinction coefficient: 0.0014 (2)

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.90858 (2)	0.20782 (14)	0.56665 (4)	0.0858 (3)
Cl2	0.96867 (2)	0.10906 (15)	0.90003 (5)	0.0939 (4)
N1	0.78571 (6)	0.0580 (3)	0.76338 (12)	0.0430 (5)
N2	0.74565 (5)	0.0416 (3)	0.78172 (11)	0.0449 (5)
H2	0.7378	0.0201	0.8342	0.054*
O1	0.83142 (5)	0.1268 (2)	0.64024 (10)	0.0538 (5)
H1	0.8097	0.1104	0.6635	0.081*
O2	0.73122 (5)	0.0679 (2)	0.63564 (10)	0.0550 (5)
O3	0.55192 (6)	0.1318 (3)	0.76671 (15)	0.0877 (7)
O4	0.28854 (6)	0.0811 (2)	0.04707 (10)	0.0605 (5)
H18	0.2830	0.0895	−0.0081	0.091*
H19	0.3050	0.1670	0.0597	0.091*

C1	0.86185 (7)	0.1190 (3)	0.70224 (14)	0.0445 (6)
C2	0.90114 (8)	0.1561 (4)	0.67682 (15)	0.0528 (6)
C3	0.93372 (8)	0.1525 (4)	0.73556 (18)	0.0636 (7)
H3	0.9597	0.1786	0.7172	0.076*
C4	0.92734 (8)	0.1095 (4)	0.82283 (17)	0.0596 (7)
C5	0.88941 (7)	0.0692 (3)	0.85008 (16)	0.0503 (6)
H5	0.8860	0.0387	0.9090	0.060*
C6	0.85555 (7)	0.0729 (3)	0.79125 (14)	0.0409 (5)
C7	0.81465 (7)	0.0354 (3)	0.82230 (14)	0.0397 (5)
C8	0.80792 (7)	−0.0152 (3)	0.91630 (14)	0.0437 (5)
H8A	0.8310	−0.0844	0.9394	0.052*
H8B	0.7840	−0.0916	0.9186	0.052*
C9	0.80230 (9)	0.1511 (4)	0.97375 (16)	0.0606 (7)
H9A	0.8275	0.2160	0.9799	0.091*
H9B	0.7939	0.1141	1.0311	0.091*
H9C	0.7819	0.2284	0.9466	0.091*
C10	0.71868 (7)	0.0615 (3)	0.71071 (14)	0.0418 (5)
C11	0.67535 (7)	0.0788 (3)	0.72990 (14)	0.0422 (5)
C12	0.66180 (7)	0.1335 (3)	0.81114 (15)	0.0505 (6)
H12	0.6807	0.1569	0.8570	0.061*
C13	0.62131 (8)	0.1540 (4)	0.82595 (17)	0.0584 (7)
H13	0.6130	0.1909	0.8812	0.070*
C14	0.59297 (8)	0.1198 (4)	0.75892 (19)	0.0593 (7)
C15	0.60576 (8)	0.0678 (4)	0.67635 (18)	0.0621 (7)
H15	0.5867	0.0458	0.6306	0.075*
C16	0.64623 (7)	0.0488 (3)	0.66187 (16)	0.0522 (6)
H16	0.6545	0.0154	0.6061	0.063*
C17	0.53716 (10)	0.1785 (6)	0.8521 (2)	0.1014 (12)
H17A	0.5465	0.0903	0.8950	0.152*
H17B	0.5080	0.1802	0.8491	0.152*
H17C	0.5472	0.2962	0.8692	0.152*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0798 (6)	0.1289 (8)	0.0498 (4)	−0.0187 (5)	0.0173 (4)	−0.0021 (4)
Cl2	0.0387 (4)	0.1699 (10)	0.0722 (5)	0.0028 (4)	−0.0099 (3)	0.0112 (5)
N1	0.0393 (11)	0.0532 (12)	0.0363 (10)	0.0011 (9)	−0.0018 (8)	0.0007 (9)
N2	0.0402 (11)	0.0603 (13)	0.0340 (10)	−0.0005 (9)	−0.0003 (8)	0.0058 (9)
O1	0.0489 (10)	0.0745 (12)	0.0378 (9)	−0.0036 (9)	−0.0019 (7)	0.0026 (8)
O2	0.0523 (10)	0.0788 (12)	0.0337 (9)	0.0002 (9)	−0.0012 (7)	0.0023 (8)
O3	0.0422 (11)	0.1199 (19)	0.1009 (17)	0.0046 (11)	0.0012 (11)	−0.0046 (14)
O4	0.0738 (12)	0.0694 (12)	0.0379 (9)	−0.0159 (9)	−0.0028 (8)	0.0036 (8)
C1	0.0480 (14)	0.0468 (13)	0.0385 (12)	0.0019 (11)	−0.0019 (10)	−0.0035 (10)
C2	0.0504 (15)	0.0648 (16)	0.0439 (13)	0.0003 (12)	0.0082 (11)	−0.0045 (12)
C3	0.0432 (15)	0.085 (2)	0.0629 (17)	0.0017 (13)	0.0084 (13)	−0.0052 (15)
C4	0.0411 (14)	0.082 (2)	0.0554 (15)	0.0071 (13)	−0.0029 (12)	−0.0018 (14)
C5	0.0442 (14)	0.0633 (16)	0.0432 (13)	0.0097 (11)	−0.0017 (11)	0.0019 (11)

C6	0.0415 (13)	0.0423 (13)	0.0388 (12)	0.0050 (10)	−0.0013 (9)	−0.0020 (10)
C7	0.0445 (13)	0.0384 (12)	0.0359 (11)	0.0038 (10)	−0.0023 (10)	−0.0016 (9)
C8	0.0425 (13)	0.0489 (13)	0.0395 (12)	0.0017 (10)	−0.0031 (10)	0.0053 (10)
C9	0.0770 (18)	0.0666 (17)	0.0383 (13)	−0.0001 (14)	0.0055 (12)	−0.0033 (12)
C10	0.0462 (13)	0.0414 (13)	0.0374 (13)	−0.0004 (10)	−0.0044 (10)	0.0002 (10)
C11	0.0426 (13)	0.0409 (12)	0.0425 (12)	−0.0026 (10)	−0.0062 (10)	0.0040 (10)
C12	0.0459 (14)	0.0614 (16)	0.0436 (13)	0.0016 (12)	−0.0049 (11)	−0.0010 (11)
C13	0.0532 (16)	0.0683 (18)	0.0541 (15)	0.0056 (13)	0.0049 (13)	0.0004 (13)
C14	0.0417 (14)	0.0641 (17)	0.0719 (18)	0.0013 (12)	0.0004 (13)	0.0059 (14)
C15	0.0477 (16)	0.0732 (19)	0.0638 (17)	−0.0041 (13)	−0.0179 (13)	−0.0016 (14)
C16	0.0525 (15)	0.0557 (15)	0.0476 (14)	−0.0007 (12)	−0.0073 (11)	−0.0025 (11)
C17	0.0529 (19)	0.139 (3)	0.114 (3)	0.009 (2)	0.0225 (19)	−0.006 (3)

Geometric parameters (Å, º)

Cl1—C2	1.736 (2)	C7—C8	1.495 (3)
Cl2—C4	1.757 (3)	C8—C9	1.519 (3)
N1—C7	1.289 (3)	C8—H8A	0.9700
N1—N2	1.364 (3)	C8—H8B	0.9700
N2—C10	1.374 (3)	C9—H9A	0.9600
N2—H2	0.8600	C9—H9B	0.9600
O1—C1	1.346 (3)	C9—H9C	0.9600
O1—H1	0.8200	C10—C11	1.473 (3)
O2—C10	1.225 (3)	C11—C12	1.384 (3)
O3—C14	1.364 (3)	C11—C16	1.395 (3)
O3—C17	1.440 (4)	C12—C13	1.369 (3)
O4—H18	0.8499	C12—H12	0.9300
O4—H19	0.8500	C13—C14	1.373 (4)
C1—C2	1.392 (3)	C13—H13	0.9300
C1—C6	1.412 (3)	C14—C15	1.388 (4)
C2—C3	1.365 (3)	C15—C16	1.366 (4)
C3—C4	1.382 (4)	C15—H15	0.9300
C3—H3	0.9300	C16—H16	0.9300
C4—C5	1.364 (3)	C17—H17A	0.9600
C5—C6	1.398 (3)	C17—H17B	0.9600
C5—H5	0.9300	C17—H17C	0.9600
C6—C7	1.470 (3)		

C7—N1—N2	122.80 (18)	C8—C9—H9A	109.5
N1—N2—C10	115.55 (18)	C8—C9—H9B	109.5
N1—N2—H2	122.2	H9A—C9—H9B	109.5
C10—N2—H2	122.2	C8—C9—H9C	109.5
C1—O1—H1	109.5	H9A—C9—H9C	109.5
C14—O3—C17	117.7 (2)	H9B—C9—H9C	109.5
H18—O4—H19	106.0	O2—C10—N2	119.8 (2)
O1—C1—C2	118.2 (2)	O2—C10—C11	123.0 (2)
O1—C1—C6	122.7 (2)	N2—C10—C11	117.13 (19)
C2—C1—C6	119.0 (2)	C12—C11—C16	117.7 (2)

C3—C2—C1	122.1 (2)	C12—C11—C10	123.3 (2)
C3—C2—Cl1	119.4 (2)	C16—C11—C10	118.9 (2)
C1—C2—Cl1	118.50 (18)	C13—C12—C11	121.9 (2)
C2—C3—C4	118.7 (2)	C13—C12—H12	119.1
C2—C3—H3	120.6	C11—C12—H12	119.1
C4—C3—H3	120.6	C12—C13—C14	119.7 (2)
C5—C4—C3	121.0 (2)	C12—C13—H13	120.1
C5—C4—Cl2	119.5 (2)	C14—C13—H13	120.1
C3—C4—Cl2	119.5 (2)	O3—C14—C13	124.8 (3)
C4—C5—C6	121.3 (2)	O3—C14—C15	115.7 (2)
C4—C5—H5	119.3	C13—C14—C15	119.5 (2)
C6—C5—H5	119.3	C16—C15—C14	120.4 (2)
C5—C6—C1	117.9 (2)	C16—C15—H15	119.8
C5—C6—C7	120.7 (2)	C14—C15—H15	119.8
C1—C6—C7	121.45 (19)	C15—C16—C11	120.7 (2)
N1—C7—C6	114.57 (19)	C15—C16—H16	119.6
N1—C7—C8	123.8 (2)	C11—C16—H16	119.6
C6—C7—C8	121.57 (18)	O3—C17—H17A	109.5
C7—C8—C9	111.64 (19)	O3—C17—H17B	109.5
C7—C8—H8A	109.3	H17A—C17—H17B	109.5
C9—C8—H8A	109.3	O3—C17—H17C	109.5
C7—C8—H8B	109.3	H17A—C17—H17C	109.5
C9—C8—H8B	109.3	H17B—C17—H17C	109.5
H8A—C8—H8B	108.0		

C7—N1—N2—C10	−177.1 (2)	C1—C6—C7—C8	179.4 (2)
O1—C1—C2—C3	179.5 (2)	N1—C7—C8—C9	−88.7 (3)
C6—C1—C2—C3	−1.2 (4)	C6—C7—C8—C9	88.0 (3)
O1—C1—C2—Cl1	−0.7 (3)	N1—N2—C10—O2	9.7 (3)
C6—C1—C2—Cl1	178.64 (18)	N1—N2—C10—C11	−168.88 (19)
C1—C2—C3—C4	0.4 (4)	O2—C10—C11—C12	−157.1 (2)
Cl1—C2—C3—C4	−179.4 (2)	N2—C10—C11—C12	21.4 (3)
C2—C3—C4—C5	0.7 (4)	O2—C10—C11—C16	19.2 (3)
C2—C3—C4—Cl2	−178.6 (2)	N2—C10—C11—C16	−162.3 (2)
C3—C4—C5—C6	−1.1 (4)	C16—C11—C12—C13	1.4 (4)
Cl2—C4—C5—C6	178.2 (2)	C10—C11—C12—C13	177.8 (2)
C4—C5—C6—C1	0.3 (4)	C11—C12—C13—C14	0.0 (4)
C4—C5—C6—C7	−177.8 (2)	C17—O3—C14—C13	−1.9 (4)
O1—C1—C6—C5	−179.9 (2)	C17—O3—C14—C15	177.7 (3)
C2—C1—C6—C5	0.8 (3)	C12—C13—C14—O3	178.4 (2)
O1—C1—C6—C7	−1.8 (3)	C12—C13—C14—C15	−1.1 (4)
C2—C1—C6—C7	178.9 (2)	O3—C14—C15—C16	−178.9 (3)
N2—N1—C7—C6	−175.44 (18)	C13—C14—C15—C16	0.7 (4)
N2—N1—C7—C8	1.4 (3)	C14—C15—C16—C11	0.8 (4)
C5—C6—C7—N1	174.4 (2)	C12—C11—C16—C15	−1.9 (4)
C1—C6—C7—N1	−3.6 (3)	C10—C11—C16—C15	−178.4 (2)
C5—C6—C7—C8	−2.6 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.77	2.494 (2)	146
N2—H2···O4^{i}	0.86	2.16	3.004 (2)	168
O4—H18···O2^{ii}	0.85	1.97	2.815 (2)	171
O4—H19···O1^{iii}	0.85	2.11	2.908 (2)	155

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