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N′-[(*E*)-2-Hydroxy-5-methoxybenzylidene]-2-methoxybenzohydrazide

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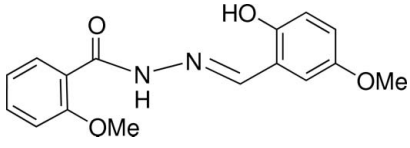
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.124; data-to-parameter ratio = 13.3.

The molecule of the title compound, $C_{16}H_{16}N_2O_4$, adopts an E conformation about the azomethine C=N double bond. The dihedral angle formed by the benzene rings is 18.88 (9)°. The molecular conformation is stabilized by an intramolecular O—H···N hydrogen bond, which forms an S(6) ring. In the crystal, the molecules are linked into chains parallel to [001] by N—H···O hydrogen bonds. The chains are further connected into a three-dimensional network by π–π stacking interactions with centroid–centroid distances of 3.6538 (10) and 3.8995 (11) Å.

Related literature

For the applications and biological activity of Schiff bases, see: Panneerselvam *et al.* (2009); Khan *et al.* (2009); Jarahpour *et al.* (2007); Pandeya *et al.* (1999). For related structures, see: Taha *et al.* (2012*a*,*b*); Lu *et al.* (2008).



Experimental

Crystal data

$C_{16}H_{16}N_2O_4$
M_r = 300.31
Monoclinic, $P2_1/c$
a = 14.5775 (13) Å
b = 11.0798 (11) Å
c = 9.5893 (9) Å
β = 99.872 (2)°
V = 1525.9 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 273 K
0.59 × 0.45 × 0.39 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.946, T_{max} = 0.964
8886 measured reflections
2767 independent reflections
2210 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.124
S = 1.04
2767 reflections
208 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N1	0.83 (2)	1.87 (2)	2.605 (2)	146.0 (19)
N2—H2A···O3^{i}	0.835 (17)	2.051 (17)	2.8258 (17)	154.2 (15)

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ5014).

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supporting information

Acta Cryst. (2012). E**68**, o3255 [doi:10.1107/S1600536812042389]

N′-[(*E*)-2-Hydroxy-5-methoxybenzylidene]-2-methoxybenzohydrazide

M. Syukri. Baharudin, Muhammad Taha, Nor Hadiani Ismail, Syed Adnan Ali Shah and Sammer Yousuf

S1. Comment

Applications of Schiff bases are reported in different fields of chemistry with a broad range of biological activities (Panneerselvam *et al.*, 2009, Khan *et al.*, 2009, Jarahpour *et al.*, 2007, Pandeya *et al.*, 1999). The title compound is a Schiff base synthesize as a part of our ongoing resaerch to study different biological activities of this medicinally important class of organic compounds.

The structure of title compound (Fig. 1) is similar to that of the previously published compound *N*′-(2-hydroxybenzylidene)-2-methoxybenzohydrazide monohydrate (Lu *et al.*, 2008), with the difference that the 2-hydroxy benzene ring is replaced by a 2-hydroxy-5-methoxy phenyl ring (C1–C6). The phenyl rings (C1–C6 and C9–C14) form an angle of 18.88 (9)°. Bond lengths and angles are similar to those observed in structurally related benzohydrazide derivatives (Taha *et al.*, 2012; Lu *et al.*, 2008). The *E* configuration of the azomethine olefinic bond is stabilized by an intramolecular O1—H1A···N1 hydrogen bond (Table 1) forming a ring of *S*(6) graph set motif. The crystal structure is stabilized by an intermolecular N2—H2A···O3 interaction forming chains running parallel to the [001] direction (Fig. 2). The chains are further linked into a three-dimensional network by $\pi\cdots\pi$ stacking interactions with centroid–centroid distances of 3.6538 (10) and 3.8995 (11) Å.

S2. Experimental

The title compound was synthesized by refluxing a mixture of 2-methoxybenzohydrazide (0.332 g, 2 mmol) and 2-hydroxy-5-methoxybenzaldehyde (0.304 g, 2 mmol) in methanol along with a catalytical amount of acetic acid for 3 hrs. The progress of reaction was monitored by TLC. After completion of reaction, the solvent was evaporated by vacuum to afford the crude product which was recrystallized by dissolving in methanol at room temperature to obtained needle-like crystals (0.504 g, 84% yield). All chemicals were purchased by Sigma Aldrich Germany.

S3. Refinement

H atoms on methyl, phenyl and methine carbon atoms were positioned geometrically with C—H = 0.96 Å (CH_3) and 0.93 Å (CH), and constrained to ride on their parent atoms with U_{iso}(H) = 1.5U_{eq}(CH_3) or 1.2U_{eq}(CH). The H atoms on the nitrogen (N–H= 0.835 (17) Å) and oxygen (O–H= 0.84 (2) Å) atoms were located in a difference Fourier map and refined isotropically. A rotating group model was applied to the methyl groups.

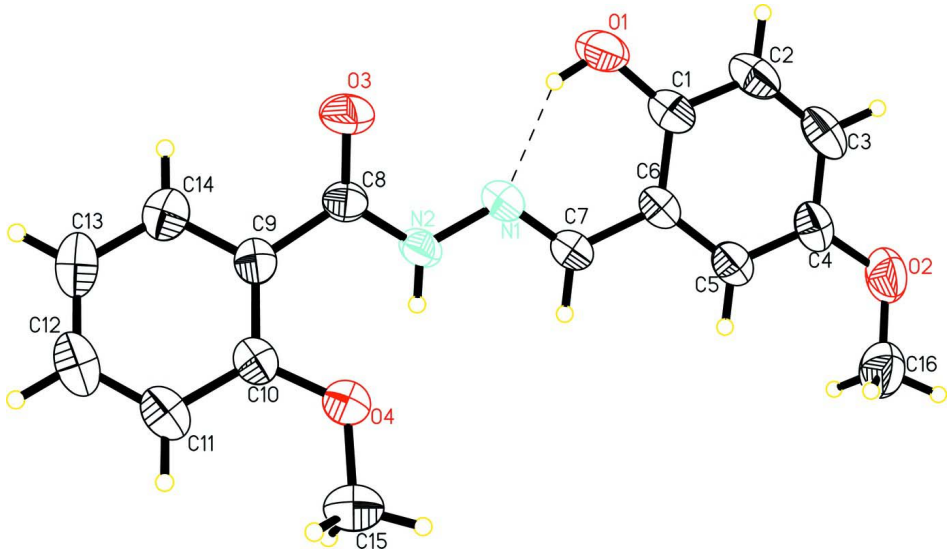

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level. The intramolecular hydrogen bond is shown as a dashed line.

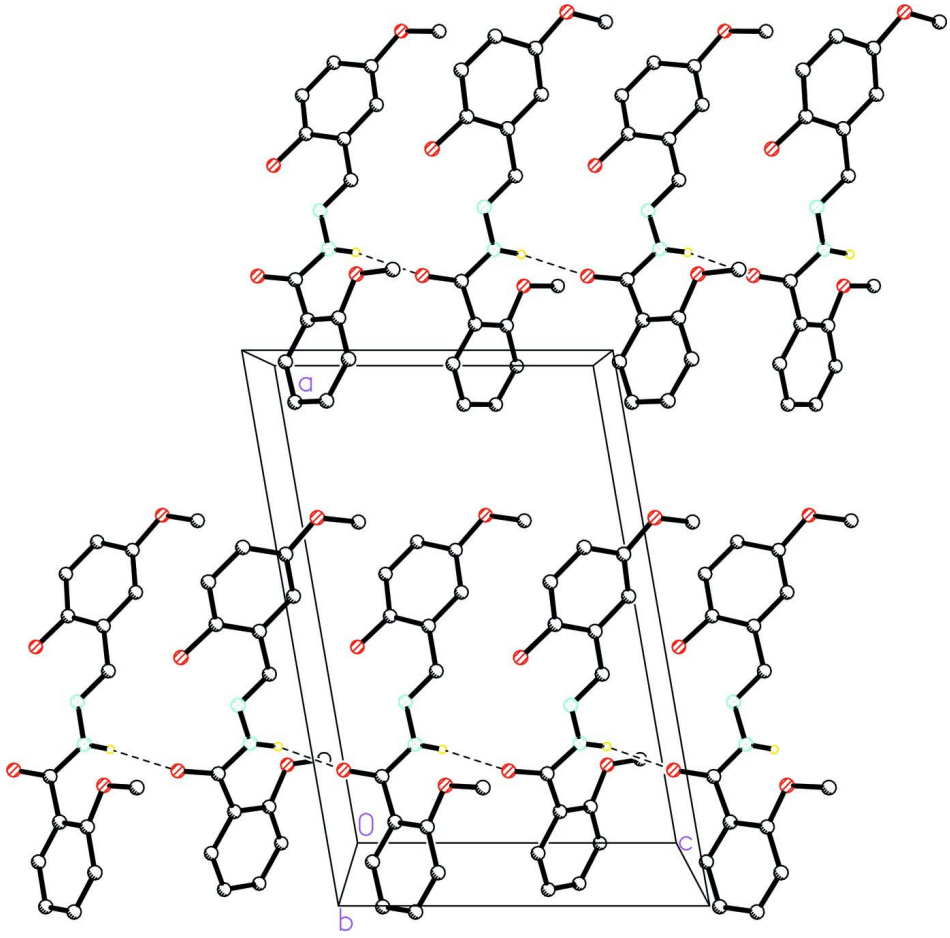

Figure 2

The crystal packing of the title compound. Only hydrogen atoms involved in hydrogen bonding (dashed lines) are shown.

N′-[(*E*)-2-Hydroxy-5-methoxybenzylidene]-2-methoxybenzohydrazide

Crystal data

$C_{16}H_{16}N_2O_4$
$M_r = 300.31$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 14.5775\ (13)$ Å
$b = 11.0798\ (11)$ Å
$c = 9.5893\ (9)$ Å
$\beta = 99.872\ (2)°$
$V = 1525.9\ (2)$ Å^3
$Z = 4$
$F(000) = 632$
$D_x = 1.307$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3190 reflections
$\theta = 2.3–25.0°$
$\mu = 0.10$ mm^{-1}
$T = 273$ K
Block, colourless
0.59 × 0.45 × 0.39 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.946$, $T_{max} = 0.964$
8886 measured reflections
2767 independent reflections
2210 reflections with $I > 2\sigma(I)$

R_{int} = 0.016
θ_{max} = 25.5°, θ_{min} = 1.4°
h = −17→16
k = −13→13
l = −9→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.124
S = 1.04
2767 reflections
208 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
$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.1784P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.41411 (11)	0.06306 (13)	0.11043 (13)	0.0918 (4)
O2	0.68838 (9)	0.10138 (14)	0.58074 (17)	0.1009 (4)
O3	0.17476 (9)	0.24072 (14)	0.01591 (12)	0.0957 (5)
O4	0.16599 (8)	0.47356 (10)	0.32496 (12)	0.0767 (3)
N1	0.31001 (9)	0.20202 (11)	0.23724 (13)	0.0618 (3)
N2	0.22889 (9)	0.26092 (12)	0.24670 (14)	0.0634 (4)
C1	0.47885 (12)	0.07530 (15)	0.22962 (18)	0.0714 (5)
C2	0.56411 (15)	0.01986 (17)	0.2354 (2)	0.0881 (6)
H2B	0.5758	−0.0250	0.1584	0.106*
C3	0.63154 (14)	0.03008 (17)	0.3527 (2)	0.0884 (6)
H3A	0.6885	−0.0083	0.3548	0.106*
C4	0.61614 (11)	0.09696 (16)	0.4685 (2)	0.0752 (5)
C5	0.53108 (11)	0.15170 (14)	0.46503 (19)	0.0692 (4)
H5A	0.5198	0.1959	0.5428	0.083*
C6	0.46152 (10)	0.14167 (13)	0.34613 (16)	0.0612 (4)
C7	0.37267 (11)	0.19998 (13)	0.34759 (17)	0.0629 (4)
H7A	0.3612	0.2364	0.4303	0.076*
C8	0.16545 (10)	0.28010 (13)	0.13077 (15)	0.0585 (4)
C9	0.08121 (10)	0.34987 (13)	0.15057 (14)	0.0578 (4)
C10	0.08163 (11)	0.44261 (14)	0.24890 (16)	0.0631 (4)
C11	−0.00098 (14)	0.50192 (17)	0.2600 (2)	0.0822 (5)

H11A	−0.0012	0.5626	0.3270	0.099*
C12	−0.08169 (14)	0.47113 (19)	0.1728 (3)	0.0922 (6)
H12A	−0.1367	0.5106	0.1818	0.111*
C13	−0.08304 (12)	0.3835 (2)	0.0728 (2)	0.0883 (6)
H13A	−0.1383	0.3641	0.0131	0.106*
C14	−0.00128 (12)	0.32362 (16)	0.06098 (18)	0.0743 (5)
H14A	−0.0019	0.2648	−0.0083	0.089*
C15	0.17013 (18)	0.5590 (2)	0.4353 (3)	0.1167 (8)
H15A	0.2339	0.5716	0.4783	0.175*
H15B	0.1357	0.5295	0.5050	0.175*
H15C	0.1437	0.6339	0.3974	0.175*
C16	0.67844 (15)	0.1759 (2)	0.6962 (3)	0.1081 (7)
H16A	0.7359	0.1775	0.7622	0.162*
H16B	0.6299	0.1448	0.7421	0.162*
H16C	0.6628	0.2563	0.6629	0.162*
H2A	0.2202 (11)	0.2826 (14)	0.3268 (18)	0.066 (5)*
H1A	0.3662 (16)	0.101 (2)	0.120 (2)	0.106 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1031 (10)	0.1066 (10)	0.0695 (8)	0.0320 (8)	0.0257 (7)	−0.0085 (7)
O2	0.0627 (8)	0.1096 (10)	0.1274 (11)	0.0180 (7)	0.0077 (8)	−0.0032 (9)
O3	0.1036 (10)	0.1328 (11)	0.0532 (7)	0.0220 (8)	0.0202 (6)	−0.0145 (7)
O4	0.0744 (8)	0.0750 (7)	0.0801 (7)	0.0077 (6)	0.0118 (6)	−0.0138 (6)
N1	0.0644 (8)	0.0624 (7)	0.0623 (8)	0.0105 (6)	0.0214 (7)	0.0013 (6)
N2	0.0642 (8)	0.0780 (8)	0.0509 (7)	0.0163 (6)	0.0180 (6)	−0.0018 (6)
C1	0.0783 (11)	0.0701 (10)	0.0719 (10)	0.0150 (8)	0.0298 (9)	0.0068 (8)
C2	0.0933 (14)	0.0870 (12)	0.0924 (13)	0.0288 (11)	0.0400 (12)	−0.0002 (10)
C3	0.0740 (12)	0.0836 (12)	0.1166 (16)	0.0274 (10)	0.0418 (12)	0.0122 (11)
C4	0.0571 (10)	0.0719 (10)	0.0989 (12)	0.0093 (8)	0.0204 (9)	0.0092 (9)
C5	0.0634 (10)	0.0658 (9)	0.0826 (11)	0.0059 (7)	0.0241 (9)	−0.0007 (8)
C6	0.0608 (9)	0.0569 (8)	0.0708 (9)	0.0071 (7)	0.0247 (8)	0.0039 (7)
C7	0.0648 (10)	0.0622 (8)	0.0654 (9)	0.0073 (7)	0.0211 (8)	−0.0030 (7)
C8	0.0666 (9)	0.0629 (8)	0.0488 (8)	−0.0017 (7)	0.0180 (7)	0.0036 (6)
C9	0.0581 (9)	0.0632 (8)	0.0539 (8)	0.0000 (7)	0.0142 (7)	0.0152 (6)
C10	0.0643 (10)	0.0622 (8)	0.0654 (9)	0.0052 (7)	0.0183 (8)	0.0104 (7)
C11	0.0774 (12)	0.0749 (11)	0.0991 (13)	0.0142 (9)	0.0293 (10)	0.0068 (9)
C12	0.0663 (12)	0.0863 (13)	0.1271 (17)	0.0149 (10)	0.0258 (12)	0.0237 (12)
C13	0.0587 (11)	0.0930 (13)	0.1090 (15)	−0.0021 (9)	0.0026 (10)	0.0306 (12)
C14	0.0737 (11)	0.0744 (10)	0.0733 (10)	−0.0056 (8)	0.0082 (9)	0.0145 (8)
C15	0.1156 (18)	0.1105 (17)	0.1212 (18)	0.0065 (13)	0.0127 (14)	−0.0508 (14)
C16	0.0801 (14)	0.1211 (18)	0.1162 (17)	0.0058 (12)	−0.0030 (12)	−0.0012 (15)

Geometric parameters (Å, º)

O1—C1	1.359 (2)	C6—C7	1.450 (2)
O1—H1A	0.84 (2)	C7—H7A	0.9300

O2—C4	1.372 (2)	C8—C9	1.491 (2)
O2—C16	1.408 (3)	C9—C14	1.384 (2)
O3—C8	1.2138 (17)	C9—C10	1.394 (2)
O4—C10	1.362 (2)	C10—C11	1.392 (2)
O4—C15	1.413 (2)	C11—C12	1.365 (3)
N1—C7	1.2740 (18)	C11—H11A	0.9300
N1—N2	1.3671 (17)	C12—C13	1.362 (3)
N2—C8	1.3356 (19)	C12—H12A	0.9300
N2—H2A	0.835 (17)	C13—C14	1.386 (3)
C1—C2	1.379 (2)	C13—H13A	0.9300
C1—C6	1.396 (2)	C14—H14A	0.9300
C2—C3	1.366 (3)	C15—H15A	0.9600
C2—H2B	0.9300	C15—H15B	0.9600
C3—C4	1.385 (3)	C15—H15C	0.9600
C3—H3A	0.9300	C16—H16A	0.9600
C4—C5	1.376 (2)	C16—H16B	0.9600
C5—C6	1.394 (2)	C16—H16C	0.9600
C5—H5A	0.9300		

C1—O1—H1A	109.1 (15)	C14—C9—C10	118.52 (15)
C4—O2—C16	117.98 (14)	C14—C9—C8	117.34 (14)
C10—O4—C15	119.23 (15)	C10—C9—C8	124.08 (14)
C7—N1—N2	117.29 (12)	O4—C10—C11	123.63 (16)
C8—N2—N1	120.27 (12)	O4—C10—C9	116.54 (13)
C8—N2—H2A	121.8 (11)	C11—C10—C9	119.75 (16)
N1—N2—H2A	117.9 (11)	C12—C11—C10	120.09 (19)
O1—C1—C2	118.72 (16)	C12—C11—H11A	120.0
O1—C1—C6	122.06 (14)	C10—C11—H11A	120.0
C2—C1—C6	119.22 (17)	C13—C12—C11	121.09 (18)
C3—C2—C1	120.82 (17)	C13—C12—H12A	119.5
C3—C2—H2B	119.6	C11—C12—H12A	119.5
C1—C2—H2B	119.6	C12—C13—C14	119.35 (18)
C2—C3—C4	120.80 (16)	C12—C13—H13A	120.3
C2—C3—H3A	119.6	C14—C13—H13A	120.3
C4—C3—H3A	119.6	C9—C14—C13	121.11 (18)
O2—C4—C5	124.81 (17)	C9—C14—H14A	119.4
O2—C4—C3	116.11 (16)	C13—C14—H14A	119.4
C5—C4—C3	119.07 (18)	O4—C15—H15A	109.5
C4—C5—C6	120.71 (16)	O4—C15—H15B	109.5
C4—C5—H5A	119.6	H15A—C15—H15B	109.5
C6—C5—H5A	119.6	O4—C15—H15C	109.5
C5—C6—C1	119.36 (14)	H15A—C15—H15C	109.5
C5—C6—C7	118.83 (14)	H15B—C15—H15C	109.5
C1—C6—C7	121.80 (15)	O2—C16—H16A	109.5
N1—C7—C6	120.96 (14)	O2—C16—H16B	109.5
N1—C7—H7A	119.5	H16A—C16—H16B	109.5
C6—C7—H7A	119.5	O2—C16—H16C	109.5
O3—C8—N2	121.93 (14)	H16A—C16—H16C	109.5

O3—C8—C9	121.66 (14)	H16B—C16—H16C	109.5
N2—C8—C9	116.39 (12)		
C7—N1—N2—C8	171.24 (14)	N1—N2—C8—O3	4.2 (2)
O1—C1—C2—C3	179.69 (17)	N1—N2—C8—C9	−177.33 (12)
C6—C1—C2—C3	−0.6 (3)	O3—C8—C9—C14	29.3 (2)
C1—C2—C3—C4	−0.4 (3)	N2—C8—C9—C14	−149.17 (14)
C16—O2—C4—C5	−6.0 (3)	O3—C8—C9—C10	−147.80 (16)
C16—O2—C4—C3	175.16 (18)	N2—C8—C9—C10	33.75 (19)
C2—C3—C4—O2	180.00 (17)	C15—O4—C10—C11	9.0 (2)
C2—C3—C4—C5	1.1 (3)	C15—O4—C10—C9	−174.19 (17)
O2—C4—C5—C6	−179.68 (15)	C14—C9—C10—O4	−173.55 (13)
C3—C4—C5—C6	−0.9 (3)	C8—C9—C10—O4	3.5 (2)
C4—C5—C6—C1	0.0 (2)	C14—C9—C10—C11	3.4 (2)
C4—C5—C6—C7	179.48 (14)	C8—C9—C10—C11	−179.56 (14)
O1—C1—C6—C5	−179.50 (15)	O4—C10—C11—C12	175.27 (16)
C2—C1—C6—C5	0.8 (2)	C9—C10—C11—C12	−1.4 (3)
O1—C1—C6—C7	1.0 (2)	C10—C11—C12—C13	−0.8 (3)
C2—C1—C6—C7	−178.75 (15)	C11—C12—C13—C14	0.9 (3)
N2—N1—C7—C6	−178.73 (13)	C10—C9—C14—C13	−3.3 (2)
C5—C6—C7—N1	173.93 (14)	C8—C9—C14—C13	179.48 (14)
C1—C6—C7—N1	−6.6 (2)	C12—C13—C14—C9	1.1 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1	0.83 (2)	1.87 (2)	2.605 (2)	146.0 (19)
N2—H2*A*···O3^{i}	0.835 (17)	2.051 (17)	2.8258 (17)	154.2 (15)

Symmetry code: (i) x, $-y+1/2$, $z+1/2$.