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(2*E*)-*N*′-[(*E*)-2-Hydroxybenzylidene]-3-phenylprop-2-enohydrazide

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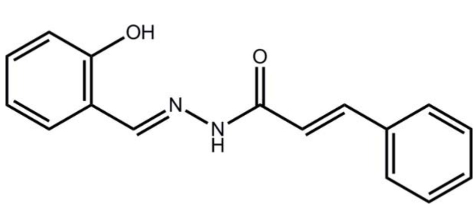
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.075; data-to-parameter ratio = 8.7.

In the non-planar title compound, $C_{16}H_{14}N_2O_2$, the dihedral angle between the phenyl rings is 16.67 (8)°. An *E* conformation is found for each of the imine [1.286 (2) Å] and ethylene [1.335 (2) Å] bonds. The amide O and H atoms are *anti*, and an intramolecular hydroxy O—H⋯N hydrogen bond is noted. The formation of N—H⋯O(hydroxy) hydrogen bonds in the crystal packing leads to helical chains along the *b* axis. Supramolecular layers in the *ab* plane are formed as the chains are linked by C—H⋯O interactions.

Related literature

For background to the biological activity of compounds with the *N*-acylhydrazone framework, (*E*)-cinnamoylhydrazone derivatives, and related structures, see: Carvalho *et al.* (2012*a*). For the synthesis, see: Carvalho *et al.* (2012*b*). For background to the data collection at the National Crystallographic Service, see: Coles & Gale (2012).



Experimental

Crystal data

$C_{16}H_{14}N_2O_2$	V = 1310.29 (12) Å^3
M_r = 266.29	Z = 4
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation
a = 24.2707 (17) Å	μ = 0.09 mm^{-1}
b = 5.1322 (2) Å	T = 100 K
c = 10.5192 (4) Å	0.19 × 0.09 × 0.03 mm

Data collection

Rigaku Saturn724+ diffractometer	5871 measured reflections
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2011)	1575 independent reflections
T_{min} = 0.878, T_{max} = 1.000	1504 reflections with $I > 2\sigma(I)$
	R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.075	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
S = 0.93	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
1575 reflections	
181 parameters	
1 restraint	

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H1o⋯N1	0.85 (2)	1.86 (2)	2.6080 (19)	147 (2)
N2—H2n⋯O1^{i}	0.88 (1)	2.05 (1)	2.9070 (19)	165 (2)
C3—H3⋯O2^{ii}	0.95	2.54	3.215 (2)	128
C7—H7⋯O2^{ii}	0.95	2.47	3.174 (2)	131

Symmetry codes: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2011); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6860).

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S1. Comment

Compounds related to the title (*E*)-cinnamoylhydrazone derivative, (I), are of interest owing to their biological activities (Carvalho *et al.*, 2012*a*). For example, (I), exhibits considerable trypanocidal activity (Carvalho *et al.*, 2012*b*). Herein, the crystal structure determination of (I) is described.

In (I), Fig. 1, there is a twist in the molecule as seen in the dihedral angle between the phenyl rings of 16.67 (8)°. The greatest deviation from a planar torsion angle is found for C9—C10—C11—C16 of 8.4 (3)°. There is an intramolecular hydroxy-O1···N2 hydrogen bond. The conformation about each of the imine [N1═C7 = 1.286 (2) Å] and ethylene [C9═C10 = 1.335 (2) Å] bonds is *E*. The amide-O and –H atoms are *anti*. The molecular structure of (I) resembles that of the unsubstituted compound (Carvalho *et al.*, 2012*a*) where the dihedral angle between terminal phenyl rings is 25.48 (12)°.

The formation of N—H···O hydrogen bonds between the amide-H and hydroxyl-O leads to helical supramolecular chains along the *b* axis, Fig. 2 and Table 1. The chains are linked into a supramolecular layer in the *ab* plane by C—H···O interactions, Fig. 3 and Table 1; the layers inter-digitate along the *c* axis, Fig. 4.

S2. Experimental

The title compound was prepared as reported (Carvalho *et al.*, 2012*b*). The sample used in the crystallographic study was grown from its EtOH solution and intensity data was collected at the National Crystallographic Service, England (Coles & Gale, 2012).

S3. Refinement

The C-bound H atoms were geometrically placed (C—H = 0.95 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C). The O– and N-bound H atoms were located from a difference map and refined with the distance restraint O—H = 0.84±0.01 and N—H = 0.88±0.01 Å, and with U_{iso}(H) = zU_{eq}(carrier atom); z = 1.5 for O and z = 1.2 for N. In the absence of significant anomalous scattering effects, 1033 Friedel pairs were averaged in the final refinement. One reflection, *i.e.* (20 0 0) was omitted from the final refinement owing to poor agreement.

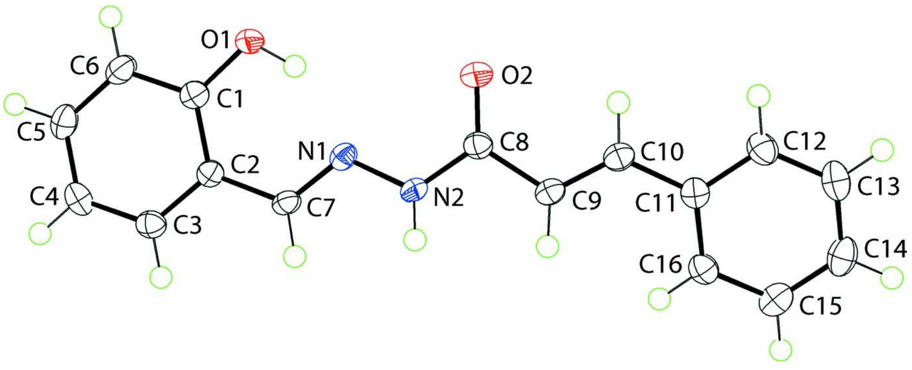

Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 70% probability level.

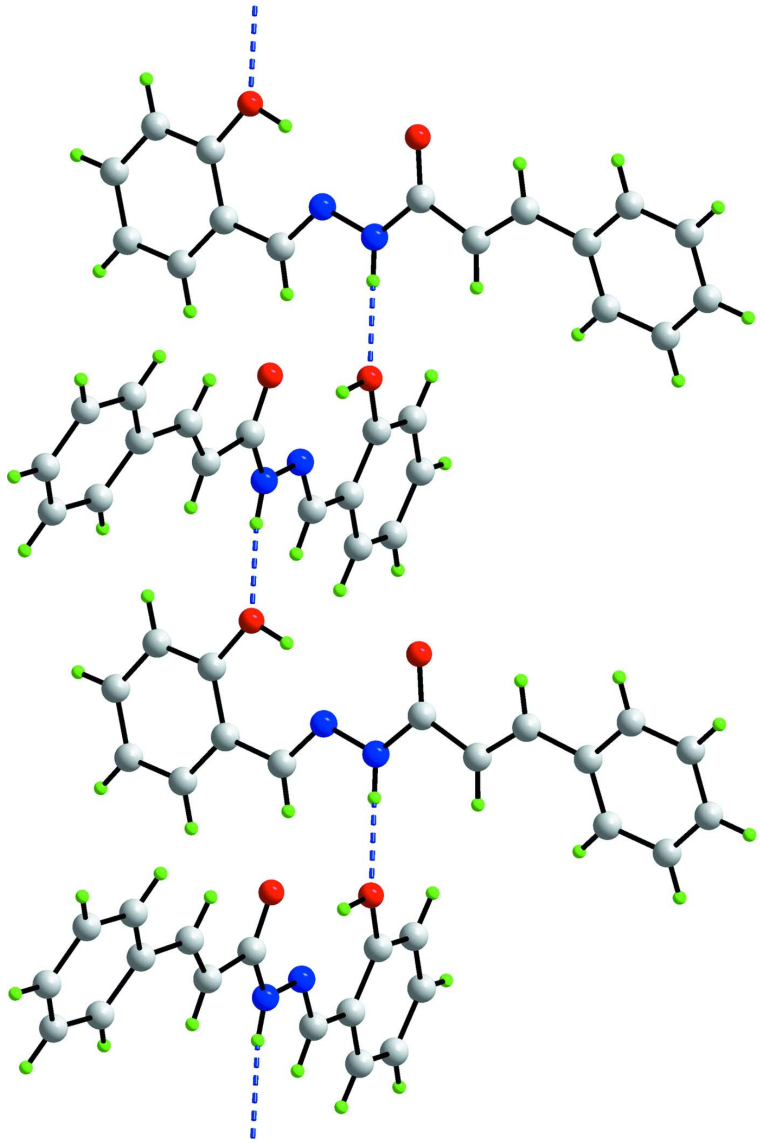

Figure 2

A view of the supramolecular helical chain along the *b* axis in (I). The N—H···O hydrogen bonds are shown as blue dashed lines.

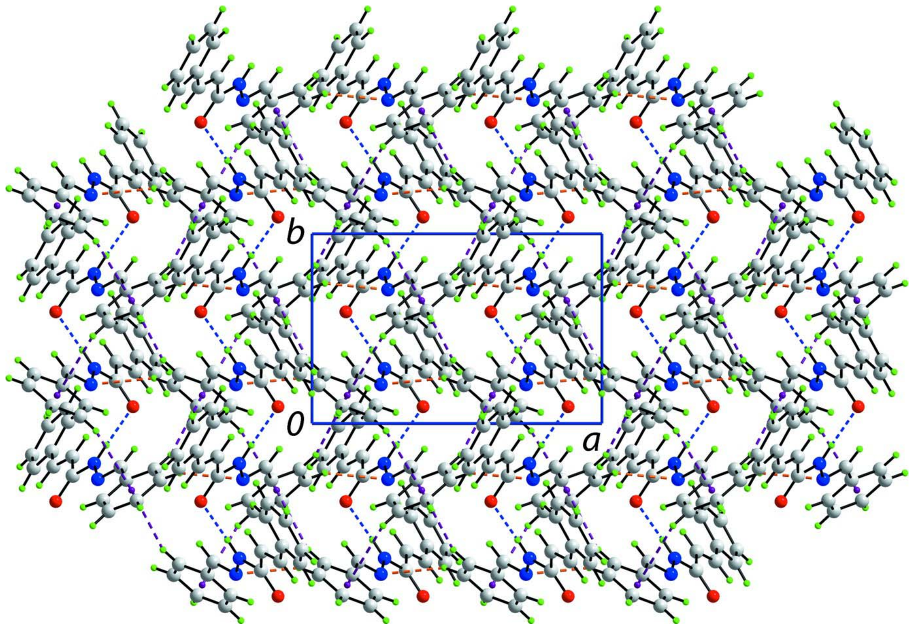

Figure 3

A view of the supramolecular layer in the *ab* plane in (I) sustained by N—H···O and interactions, shown as blue and orange dashed lines, respectively.

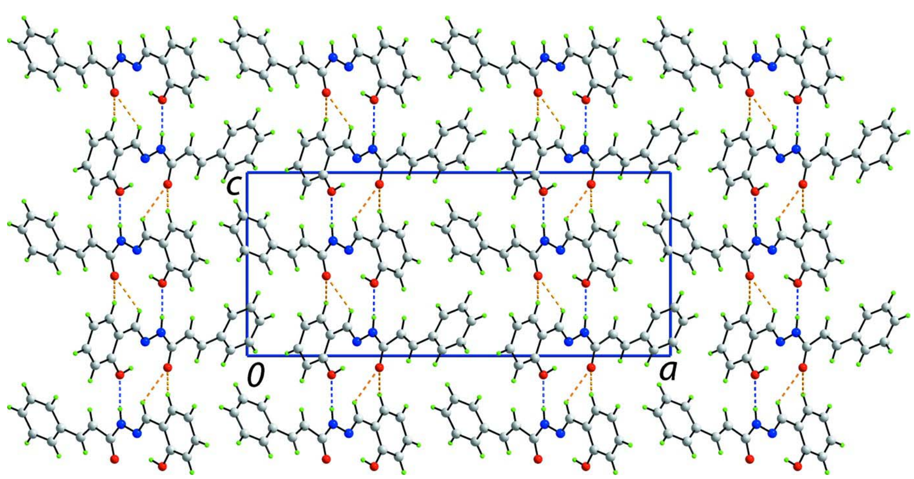

Figure 4

A view in projection down the *b* axis of the unit-cell contents for (I) showing the inter-digitation of layers. The N—H···O and C—H···O interactions are shown as blue and orange dashed lines, respectively.

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Crystal data

$C_{16}H_{14}N_2O_2$
M_r = 266.29
Orthorhombic, *Pna*2_1
Hall symbol: P 2c -2n
a = 24.2707 (17) Å
b = 5.1322 (2) Å
c = 10.5192 (4) Å
V = 1310.29 (12) Å^3
Z = 4
F(000) = 560
D_x = 1.350 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5659 reflections
θ = 3.4–27.5°

μ = 0.09 mm^{-1}
T = 100 K

Plate, colourless
0.19 × 0.09 × 0.03 mm

Data collection

Rigaku Saturn724+ diffractometer
Radiation source: Rotating Anode
Confocal monochromator
Detector resolution: 28.5714 pixels mm^{-1}
profile data from ω–scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2011)
T_{min} = 0.878, T_{max} = 1.000

5871 measured reflections
1575 independent reflections
1504 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.5°, θ_{min} = 3.4°
$h = -30 \rightarrow 31$
$k = -6 \rightarrow 6$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.075
S = 0.93
1575 reflections
181 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.3579P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Absolute structure: nd

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
O1	0.69975 (5)	0.5777 (2)	−0.10431 (12)	0.0192 (3)
H1O	0.7247 (7)	0.671 (4)	−0.071 (2)	0.029*
O2	0.81273 (5)	1.1490 (3)	−0.06463 (13)	0.0232 (3)
N1	0.75962 (6)	0.7782 (3)	0.07685 (14)	0.0150 (3)
N2	0.79692 (6)	0.9517 (3)	0.12637 (14)	0.0158 (3)
H2N	0.7997 (9)	0.959 (4)	0.2096 (10)	0.019*
C1	0.67996 (7)	0.4128 (3)	−0.01381 (17)	0.0157 (3)
C2	0.69722 (7)	0.4288 (3)	0.11420 (17)	0.0140 (3)
C3	0.67560 (7)	0.2498 (3)	0.20137 (17)	0.0171 (4)
H3	0.6869	0.2577	0.2877	0.021*
C4	0.63806 (7)	0.0613 (3)	0.16428 (19)	0.0187 (4)
H4	0.6241	−0.0602	0.2244	0.022*

C5	0.62097 (7)	0.0517 (3)	0.03833 (19)	0.0201 (4)
H5	0.5947	−0.0753	0.0128	0.024*
C6	0.64170 (7)	0.2249 (3)	−0.05053 (18)	0.0197 (4)
H6	0.6298	0.2157	−0.1365	0.024*
C7	0.73698 (7)	0.6215 (3)	0.15666 (17)	0.0150 (3)
H7	0.7463	0.6319	0.2442	0.018*
C8	0.82227 (7)	1.1293 (3)	0.04921 (17)	0.0156 (3)
C9	0.86435 (7)	1.2853 (3)	0.11785 (17)	0.0161 (3)
H9	0.8711	1.2542	0.2055	0.019*
C10	0.89272 (7)	1.4693 (3)	0.05648 (17)	0.0170 (3)
H10	0.8815	1.5065	−0.0281	0.020*
C11	0.93932 (7)	1.6197 (3)	0.10555 (17)	0.0159 (3)
C12	0.96062 (7)	1.8241 (3)	0.03252 (19)	0.0203 (4)
H12	0.9442	1.8652	−0.0469	0.024*
C13	1.00558 (8)	1.9677 (4)	0.0748 (2)	0.0226 (4)
H13	1.0196	2.1061	0.0242	0.027*
C14	1.03004 (7)	1.9097 (4)	0.19034 (19)	0.0225 (4)
H14	1.0606	2.0087	0.2193	0.027*
C15	1.00967 (7)	1.7064 (4)	0.26359 (18)	0.0210 (4)
H15	1.0266	1.6658	0.3426	0.025*
C16	0.96482 (7)	1.5624 (3)	0.22228 (18)	0.0177 (3)
H16	0.9512	1.4238	0.2732	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0245 (6)	0.0213 (6)	0.0118 (6)	−0.0067 (5)	−0.0012 (5)	−0.0003 (5)
O2	0.0293 (7)	0.0274 (7)	0.0130 (6)	−0.0075 (5)	−0.0028 (6)	−0.0001 (6)
N1	0.0147 (6)	0.0154 (6)	0.0149 (6)	−0.0003 (5)	−0.0009 (5)	−0.0031 (6)
N2	0.0175 (7)	0.0183 (7)	0.0115 (7)	−0.0035 (5)	−0.0015 (6)	−0.0028 (6)
C1	0.0148 (7)	0.0167 (7)	0.0154 (8)	0.0021 (6)	0.0008 (6)	−0.0019 (7)
C2	0.0136 (7)	0.0144 (7)	0.0141 (8)	0.0011 (6)	0.0011 (6)	−0.0023 (7)
C3	0.0170 (8)	0.0190 (8)	0.0154 (9)	0.0017 (6)	0.0011 (7)	−0.0009 (7)
C4	0.0189 (8)	0.0156 (8)	0.0215 (9)	−0.0003 (6)	0.0054 (7)	0.0021 (7)
C5	0.0175 (8)	0.0179 (8)	0.0248 (9)	−0.0029 (6)	0.0001 (7)	−0.0049 (7)
C6	0.0189 (8)	0.0225 (8)	0.0177 (8)	−0.0014 (7)	−0.0032 (7)	−0.0041 (8)
C7	0.0150 (7)	0.0182 (8)	0.0118 (7)	0.0005 (6)	−0.0007 (6)	−0.0029 (7)
C8	0.0159 (7)	0.0166 (8)	0.0143 (8)	0.0010 (6)	0.0009 (6)	−0.0013 (7)
C9	0.0167 (7)	0.0174 (8)	0.0141 (7)	0.0008 (6)	−0.0010 (6)	−0.0024 (7)
C10	0.0175 (8)	0.0176 (8)	0.0158 (8)	0.0015 (6)	−0.0009 (7)	−0.0018 (7)
C11	0.0148 (7)	0.0148 (7)	0.0180 (8)	0.0017 (6)	0.0028 (7)	−0.0019 (7)
C12	0.0212 (8)	0.0180 (8)	0.0217 (9)	0.0004 (6)	0.0004 (7)	0.0039 (7)
C13	0.0207 (9)	0.0160 (8)	0.0312 (10)	−0.0016 (6)	0.0038 (8)	0.0015 (8)
C14	0.0175 (8)	0.0189 (8)	0.0311 (11)	−0.0004 (7)	0.0009 (7)	−0.0060 (8)
C15	0.0178 (8)	0.0230 (9)	0.0223 (9)	0.0025 (7)	−0.0015 (7)	−0.0046 (8)
C16	0.0173 (7)	0.0174 (8)	0.0182 (8)	0.0010 (6)	0.0030 (7)	−0.0001 (7)

Geometric parameters (Å, º)

O1—C1	1.361 (2)	C7—H7	0.9500
O1—H1O	0.848 (10)	C8—C9	1.485 (2)
O2—C8	1.224 (2)	C9—C10	1.335 (2)
N1—C7	1.286 (2)	C9—H9	0.9500
N1—N2	1.3727 (19)	C10—C11	1.463 (2)
N2—C8	1.367 (2)	C10—H10	0.9500
N2—H2N	0.879 (10)	C11—C12	1.399 (2)
C1—C6	1.393 (2)	C11—C16	1.406 (2)
C1—C2	1.413 (3)	C12—C13	1.390 (3)
C2—C3	1.400 (2)	C12—H12	0.9500
C2—C7	1.452 (2)	C13—C14	1.385 (3)
C3—C4	1.385 (2)	C13—H13	0.9500
C3—H3	0.9500	C14—C15	1.388 (3)
C4—C5	1.389 (3)	C14—H14	0.9500
C4—H4	0.9500	C15—C16	1.386 (2)
C5—C6	1.385 (3)	C15—H15	0.9500
C5—H5	0.9500	C16—H16	0.9500
C6—H6	0.9500		
C1—O1—H1O	108.1 (18)	O2—C8—C9	124.15 (16)
C7—N1—N2	116.09 (15)	N2—C8—C9	112.36 (15)
C8—N2—N1	120.28 (15)	C10—C9—C8	120.06 (16)
C8—N2—H2N	122.0 (15)	C10—C9—H9	120.0
N1—N2—H2N	117.1 (15)	C8—C9—H9	120.0
O1—C1—C6	118.13 (17)	C9—C10—C11	126.95 (16)
O1—C1—C2	121.73 (15)	C9—C10—H10	116.5
C6—C1—C2	120.14 (17)	C11—C10—H10	116.5
C3—C2—C1	118.36 (15)	C12—C11—C16	118.26 (16)
C3—C2—C7	119.64 (16)	C12—C11—C10	119.17 (16)
C1—C2—C7	121.99 (16)	C16—C11—C10	122.55 (16)
C4—C3—C2	121.36 (17)	C13—C12—C11	120.80 (18)
C4—C3—H3	119.3	C13—C12—H12	119.6
C2—C3—H3	119.3	C11—C12—H12	119.6
C3—C4—C5	119.33 (17)	C14—C13—C12	120.23 (18)
C3—C4—H4	120.3	C14—C13—H13	119.9
C5—C4—H4	120.3	C12—C13—H13	119.9
C6—C5—C4	120.84 (17)	C13—C14—C15	119.73 (17)
C6—C5—H5	119.6	C13—C14—H14	120.1
C4—C5—H5	119.6	C15—C14—H14	120.1
C5—C6—C1	119.96 (17)	C16—C15—C14	120.44 (17)
C5—C6—H6	120.0	C16—C15—H15	119.8
C1—C6—H6	120.0	C14—C15—H15	119.8
N1—C7—C2	120.60 (16)	C15—C16—C11	120.54 (17)
N1—C7—H7	119.7	C15—C16—H16	119.7
C2—C7—H7	119.7	C11—C16—H16	119.7
O2—C8—N2	123.43 (16)		

C7—N1—N2—C8	−178.71 (15)	N1—N2—C8—O2	1.8 (3)
O1—C1—C2—C3	−179.07 (15)	N1—N2—C8—C9	−175.41 (14)
C6—C1—C2—C3	1.0 (2)	O2—C8—C9—C10	3.3 (3)
O1—C1—C2—C7	0.0 (2)	N2—C8—C9—C10	−179.52 (15)
C6—C1—C2—C7	−179.97 (16)	C8—C9—C10—C11	−172.84 (16)
C1—C2—C3—C4	−0.2 (2)	C9—C10—C11—C12	−173.45 (17)
C7—C2—C3—C4	−179.29 (15)	C9—C10—C11—C16	8.4 (3)
C2—C3—C4—C5	−0.8 (3)	C16—C11—C12—C13	−0.4 (3)
C3—C4—C5—C6	1.1 (3)	C10—C11—C12—C13	−178.64 (16)
C4—C5—C6—C1	−0.4 (3)	C11—C12—C13—C14	0.1 (3)
O1—C1—C6—C5	179.34 (15)	C12—C13—C14—C15	0.4 (3)
C2—C1—C6—C5	−0.7 (3)	C13—C14—C15—C16	−0.4 (3)
N2—N1—C7—C2	−179.55 (14)	C14—C15—C16—C11	0.1 (3)
C3—C2—C7—N1	176.11 (15)	C12—C11—C16—C15	0.3 (2)
C1—C2—C7—N1	−2.9 (2)	C10—C11—C16—C15	178.50 (16)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···N1	0.85 (2)	1.86 (2)	2.6080 (19)	147 (2)
N2—H2n···O1^{i}	0.88 (1)	2.05 (1)	2.9070 (19)	165 (2)
C3—H3···O2^{ii}	0.95	2.54	3.215 (2)	128
C7—H7···O2^{ii}	0.95	2.47	3.174 (2)	131

Symmetry codes: (i) −*x*+3/2, *y*+1/2, *z*+1/2; (ii) −*x*+3/2, *y*−1/2, *z*+1/2.