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(2,4-Dimethoxybenzylidene)-2-hydroxybenzohydrazide ethanol solvate

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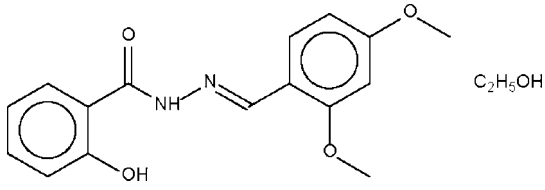
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.047; wR factor = 0.118; data-to-parameter ratio = 16.0.

In the planar title molecule, $C_{16}H_{16}N_2O_4\cdot C_2H_6O$, the planar Schiff base molecule is linked to the ethanol solvent molecule by a hydroxy–amide hydrogen bond. The hydroxy group of the ethanol molecule is a hydrogen-bond donor to the double-bonded N atom of an adjacent Sciff base, pairs of interactions taking place across a center of symmetry and giving rise to a hydrogen-bonded dimer.

Related literature

For the crystal structures of other substituted benzylidene-2-hydroxybenzohydrazides, see: Li (2007); Liang *et al.* (2005); Luo (2007); Ma *et al.* (2005); Pan & Yang (2005*a*,*b*,*c*); Qiu *et al.* (2006); Shao *et al.* (2004); Wang *et al.* (2007); Xu & Liu (2006); Yang (2006); Yang & Pan (2004, 2005*a*,*b*); Zhang *et al.* (2006).



Experimental

Crystal data

$C_{16}H_{16}N_2O_4\cdot C_2H_6O$
M_r = 346.38
Monoclinic, $P2_1/n$
a = 7.7909 (2) Å
b = 18.0539 (6) Å
c = 12.0001 (4) Å
β = 93.803 (2)°
V = 1684.17 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 (2) K
0.20 × 0.15 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: none
13796 measured reflections
3853 independent reflections
2575 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.118
S = 1.03
3853 reflections
241 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1o···O2	0.86 (1)	1.74 (2)	2.528 (2)	151 (2)
O5—H5o···N2^{i}	0.85 (1)	2.07 (1)	2.847 (2)	152 (2)
N1—H1n···O5	0.86 (1)	2.09 (1)	2.894 (2)	157 (2)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2239).

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supporting information

Acta Cryst. (2008). E**64**, o961 [doi:10.1107/S1600536808011768]

(2,4-Dimethoxybenzylidene)-2-hydroxybenzohydrazide ethanol solvate

Wagee A. Yehye, Azhar Ariffin and Seik Weng Ng

S1. Comment

The crystal structures of a number of substituted benzylidene-2-hydroxybenzohydrazides have been reported (Li, 2007; Liang *et al.*, 2005; Luo, 2007; Ma *et al.*, 2005; Pan & Yang, 2005*a*,*b*,*c*; Qiu *et al.*, 2006; Shao *et al.*, 2004; Wang *et al.*, 2007; Xu & Liu, 2006; Yang, 2006; Yang & Pan, 2004, 2005*a*,*b*; Zhang *et al.*, 2006.

The 2,4-dimethoxy derivative crystallizes as an ethanol solvate (Scheme I, Fig. 1). The planar molecule of $C_{16}H_{16}N_2O_4$ is linked to the ethanol molecule by an amido···hydroxy$_{ethanol}$ hydrogen bond [N–H···O 2.894 (2) Å]. The hydroxy unit of the ethanol molecule is a hydrogen-bond donor site to the double-bond nitrogen atom of an adjacent Sciff base [O–H···N 2.847 (2) Å], this interaction across a center of symmetry giving rise to a hydrogen-bonded dimer (Fig. 2).

S2. Experimental

2-Hydroxybenzohydrazide (0.60 g, 4 mmol) and 2,4-dimethoxybenzaldehyde (0.66 g, 4 mmol) were heated in ethanol (30 ml) for 2 h. The solvent was removed by evaporation and the product recrystallized from ethanol.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2–1.5 *U*(C).

The oxygen- and nitrogen-bound H-atoms were located in a difference Fouier map, and were refined with a distance restraint (O–H = N–H 0.85 Å); their temperature factors were freely refined.

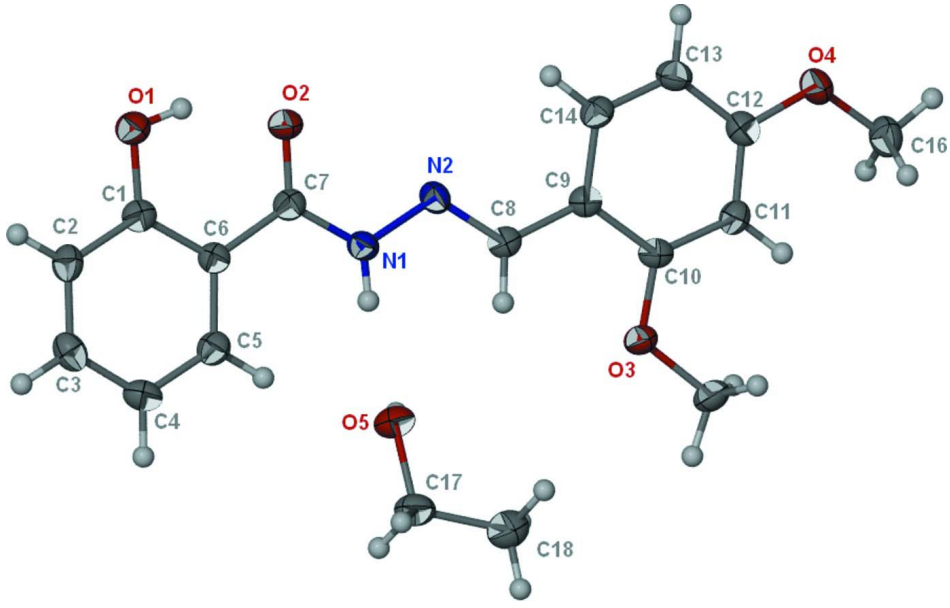

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{16}H_{16}N_2O_4{\cdot}C_2H_6O$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radiius.

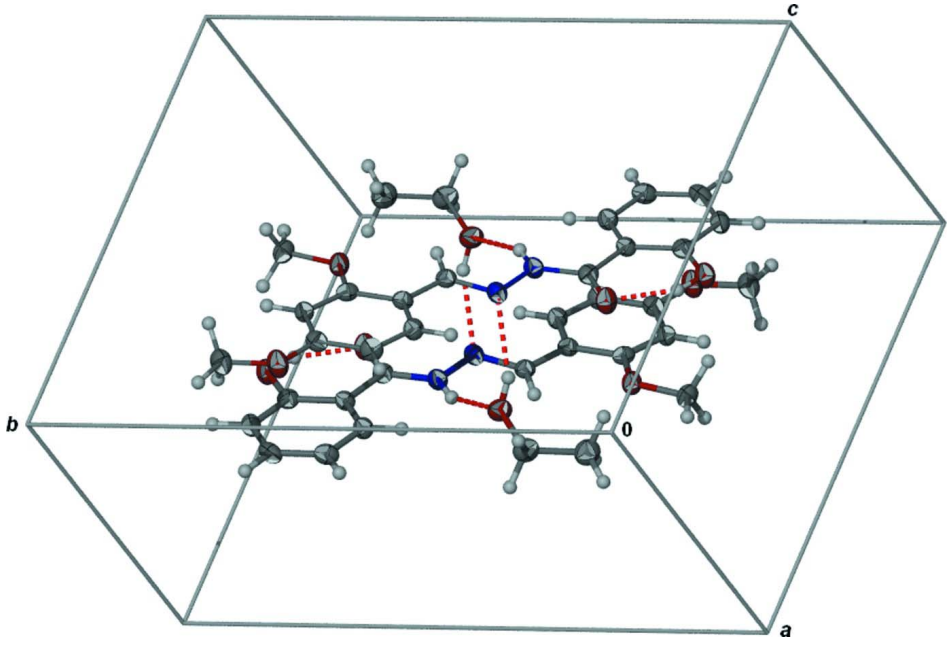

Figure 2

Hydrogen-bonded dimeric structure.

(2,4-Dimethoxybenzylidene)-2-hydroxybenzohydrazide ethanol solvate

Crystal data

$C_{16}H_{16}N_2O_4{\cdot}C_2H_6O$
M_r = 346.38

Monoclinic, $P2_1/n$
Hall symbol: -P 2yn

a = 7.7909 (2) Å
b = 18.0539 (6) Å
c = 12.0001 (4) Å
β = 93.803 (2)°
V = 1684.17 (9) Å^3
Z = 4
F(000) = 736
D_x = 1.366 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1895 reflections
θ = 2.8–25.5°
μ = 0.10 mm^{-1}
T = 100 K
Prism, colorless
0.20 × 0.15 × 0.15 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
13796 measured reflections
3853 independent reflections

2575 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 27.5°, θ_{min} = 2.0°
h = −10→10
k = −20→23
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.118
S = 1.03
3853 reflections
241 parameters
3 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
$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 0.175P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.64682 (15)	0.22307 (7)	0.69448 (11)	0.0248 (3)
O2	0.48542 (15)	0.30514 (7)	0.55256 (10)	0.0253 (3)
O3	0.09164 (14)	0.62457 (7)	0.44328 (10)	0.0224 (3)
O4	−0.15888 (16)	0.55131 (7)	0.07909 (10)	0.0280 (3)
O5	0.45339 (16)	0.56902 (7)	0.68732 (11)	0.0256 (3)
N1	0.40917 (17)	0.42200 (9)	0.59366 (12)	0.0191 (3)
N2	0.32044 (17)	0.42928 (8)	0.48948 (11)	0.0195 (3)
C1	0.6546 (2)	0.28297 (10)	0.76338 (14)	0.0199 (4)
C2	0.7381 (2)	0.27412 (10)	0.86870 (14)	0.0219 (4)
H2	0.7858	0.2275	0.8904	0.026*
C3	0.7514 (2)	0.33316 (11)	0.94120 (14)	0.0231 (4)
H3	0.8073	0.3266	1.0133	0.028*
C4	0.6847 (2)	0.40220 (10)	0.91101 (14)	0.0230 (4)
H4	0.6962	0.4428	0.9614	0.028*
C5	0.6014 (2)	0.41094 (10)	0.80671 (14)	0.0211 (4)
H5	0.5560	0.4581	0.7856	0.025*
C6	0.5825 (2)	0.35172 (10)	0.73132 (14)	0.0180 (4)
C7	0.4908 (2)	0.35796 (10)	0.61929 (14)	0.0192 (4)

C8	0.22902 (19)	0.48872 (10)	0.47864 (14)	0.0188 (4)
H8	0.2277	0.5226	0.5391	0.023*
C9	0.1282 (2)	0.50446 (10)	0.37526 (14)	0.0185 (4)
C10	0.0552 (2)	0.57510 (10)	0.35888 (14)	0.0189 (4)
C11	−0.0433 (2)	0.59274 (10)	0.26156 (14)	0.0197 (4)
H11	−0.0930	0.6405	0.2519	0.024*
C12	−0.0677 (2)	0.53942 (10)	0.17899 (14)	0.0213 (4)
C13	0.0016 (2)	0.46857 (10)	0.19353 (15)	0.0227 (4)
H13	−0.0173	0.4321	0.1369	0.027*
C14	0.0973 (2)	0.45200 (10)	0.29057 (14)	0.0205 (4)
H14	0.1438	0.4036	0.3004	0.025*
C15	0.0125 (2)	0.69592 (10)	0.43449 (15)	0.0240 (4)
H15A	0.0456	0.7250	0.5015	0.036*
H15B	−0.1128	0.6902	0.4275	0.036*
H15C	0.0505	0.7215	0.3685	0.036*
C16	−0.2167 (2)	0.62483 (11)	0.05390 (16)	0.0281 (4)
H16A	−0.2707	0.6264	−0.0222	0.042*
H16B	−0.1184	0.6588	0.0597	0.042*
H16C	−0.3006	0.6398	0.1069	0.042*
C17	0.4115 (2)	0.62589 (11)	0.76362 (15)	0.0278 (4)
H17A	0.5186	0.6506	0.7929	0.033*
H17B	0.3567	0.6033	0.8275	0.033*
C18	0.2921 (2)	0.68255 (11)	0.70951 (16)	0.0310 (5)
H18A	0.2608	0.7187	0.7655	0.046*
H18B	0.1881	0.6579	0.6775	0.046*
H18C	0.3496	0.7079	0.6502	0.046*
H1O	0.594 (3)	0.2375 (13)	0.6331 (12)	0.054 (7)*
H5O	0.518 (2)	0.5858 (13)	0.6389 (15)	0.052 (7)*
H1N	0.410 (3)	0.4596 (8)	0.6371 (14)	0.036 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0311 (7)	0.0185 (7)	0.0241 (7)	0.0015 (5)	−0.0021 (6)	−0.0001 (6)
O2	0.0328 (7)	0.0185 (7)	0.0236 (7)	0.0024 (5)	−0.0045 (5)	−0.0035 (6)
O3	0.0291 (6)	0.0170 (7)	0.0208 (7)	0.0034 (5)	−0.0009 (5)	−0.0022 (5)
O4	0.0342 (7)	0.0261 (8)	0.0225 (7)	0.0032 (6)	−0.0077 (5)	0.0004 (6)
O5	0.0313 (7)	0.0212 (7)	0.0247 (7)	0.0003 (6)	0.0063 (6)	−0.0036 (6)
N1	0.0227 (7)	0.0181 (8)	0.0163 (8)	−0.0002 (6)	−0.0007 (6)	−0.0006 (7)
N2	0.0206 (7)	0.0205 (8)	0.0169 (8)	−0.0006 (6)	−0.0012 (6)	0.0010 (7)
C1	0.0190 (8)	0.0193 (10)	0.0218 (9)	−0.0023 (7)	0.0033 (7)	−0.0003 (8)
C2	0.0212 (8)	0.0210 (10)	0.0233 (10)	0.0009 (7)	0.0000 (7)	0.0044 (8)
C3	0.0224 (8)	0.0285 (11)	0.0183 (9)	−0.0043 (8)	0.0001 (7)	0.0050 (8)
C4	0.0263 (9)	0.0235 (10)	0.0194 (9)	−0.0042 (7)	0.0031 (7)	−0.0034 (8)
C5	0.0221 (8)	0.0181 (10)	0.0232 (10)	−0.0003 (7)	0.0022 (7)	0.0017 (8)
C6	0.0192 (8)	0.0169 (10)	0.0181 (9)	−0.0020 (7)	0.0018 (6)	0.0009 (7)
C7	0.0187 (8)	0.0176 (10)	0.0214 (9)	−0.0012 (7)	0.0024 (7)	−0.0001 (8)
C8	0.0190 (8)	0.0181 (9)	0.0198 (9)	−0.0010 (7)	0.0044 (7)	0.0001 (8)

C9	0.0191 (8)	0.0187 (9)	0.0179 (9)	−0.0014 (7)	0.0032 (6)	0.0005 (8)
C10	0.0181 (8)	0.0193 (10)	0.0196 (9)	−0.0026 (7)	0.0043 (7)	−0.0026 (8)
C11	0.0195 (8)	0.0179 (10)	0.0218 (9)	0.0012 (7)	0.0027 (7)	0.0028 (8)
C12	0.0210 (8)	0.0228 (10)	0.0200 (9)	−0.0026 (7)	0.0009 (7)	0.0018 (8)
C13	0.0253 (9)	0.0206 (10)	0.0218 (10)	−0.0024 (7)	−0.0006 (7)	−0.0034 (8)
C14	0.0217 (8)	0.0158 (9)	0.0240 (10)	−0.0014 (7)	0.0023 (7)	0.0007 (8)
C15	0.0268 (9)	0.0187 (10)	0.0267 (10)	0.0036 (7)	0.0032 (7)	−0.0032 (8)
C16	0.0288 (9)	0.0294 (11)	0.0254 (10)	0.0027 (8)	−0.0031 (8)	0.0047 (9)
C17	0.0319 (10)	0.0270 (11)	0.0245 (10)	0.0004 (8)	0.0026 (8)	−0.0066 (9)
C18	0.0323 (10)	0.0270 (12)	0.0339 (11)	0.0002 (8)	0.0040 (8)	−0.0023 (9)

Geometric parameters (Å, º)

O1—C1	1.360 (2)	C8—C9	1.452 (2)
O1—H1O	0.859 (9)	C8—H8	0.9500
O2—C7	1.244 (2)	C9—C14	1.398 (2)
O3—C10	1.366 (2)	C9—C10	1.405 (2)
O3—C15	1.429 (2)	C10—C11	1.391 (2)
O4—C12	1.369 (2)	C11—C12	1.385 (2)
O4—C16	1.428 (2)	C11—H11	0.9500
O5—C17	1.428 (2)	C12—C13	1.395 (2)
O5—H5O	0.848 (10)	C13—C14	1.374 (2)
N1—C7	1.345 (2)	C13—H13	0.9500
N1—N2	1.3941 (19)	C14—H14	0.9500
N1—H1N	0.855 (9)	C15—H15A	0.9800
N2—C8	1.290 (2)	C15—H15B	0.9800
C1—C2	1.392 (2)	C15—H15C	0.9800
C1—C6	1.405 (2)	C16—H16A	0.9800
C2—C3	1.375 (2)	C16—H16B	0.9800
C2—H2	0.9500	C16—H16C	0.9800
C3—C4	1.389 (3)	C17—C18	1.501 (3)
C3—H3	0.9500	C17—H17A	0.9900
C4—C5	1.380 (2)	C17—H17B	0.9900
C4—H4	0.9500	C18—H18A	0.9800
C5—C6	1.402 (2)	C18—H18B	0.9800
C5—H5	0.9500	C18—H18C	0.9800
C6—C7	1.485 (2)		

C1—O1—H1O	106.2 (16)	C11—C10—C9	121.39 (16)
C10—O3—C15	117.86 (13)	C12—C11—C10	118.89 (16)
C12—O4—C16	117.97 (14)	C12—C11—H11	120.6
C17—O5—H5O	110.7 (17)	C10—C11—H11	120.6
C7—N1—N2	119.00 (15)	O4—C12—C11	123.77 (16)
C7—N1—H1N	123.9 (14)	O4—C12—C13	115.29 (16)
N2—N1—H1N	117.1 (14)	C11—C12—C13	120.94 (16)
C8—N2—N1	113.99 (14)	C14—C13—C12	119.31 (17)
O1—C1—C2	117.39 (16)	C14—C13—H13	120.3
O1—C1—C6	122.35 (15)	C12—C13—H13	120.3

C2—C1—C6	120.27 (16)	C13—C14—C9	121.74 (17)
C3—C2—C1	119.77 (17)	C13—C14—H14	119.1
C3—C2—H2	120.1	C9—C14—H14	119.1
C1—C2—H2	120.1	O3—C15—H15A	109.5
C2—C3—C4	121.28 (16)	O3—C15—H15B	109.5
C2—C3—H3	119.4	H15A—C15—H15B	109.5
C4—C3—H3	119.4	O3—C15—H15C	109.5
C5—C4—C3	118.97 (17)	H15A—C15—H15C	109.5
C5—C4—H4	120.5	H15B—C15—H15C	109.5
C3—C4—H4	120.5	O4—C16—H16A	109.5
C4—C5—C6	121.40 (17)	O4—C16—H16B	109.5
C4—C5—H5	119.3	H16A—C16—H16B	109.5
C6—C5—H5	119.3	O4—C16—H16C	109.5
C5—C6—C1	118.29 (15)	H16A—C16—H16C	109.5
C5—C6—C7	123.38 (16)	H16B—C16—H16C	109.5
C1—C6—C7	118.32 (15)	O5—C17—C18	111.97 (15)
O2—C7—N1	121.10 (16)	O5—C17—H17A	109.2
O2—C7—C6	121.22 (15)	C18—C17—H17A	109.2
N1—C7—C6	117.66 (15)	O5—C17—H17B	109.2
N2—C8—C9	120.86 (16)	C18—C17—H17B	109.2
N2—C8—H8	119.6	H17A—C17—H17B	107.9
C9—C8—H8	119.6	C17—C18—H18A	109.5
C14—C9—C10	117.70 (15)	C17—C18—H18B	109.5
C14—C9—C8	123.06 (16)	H18A—C18—H18B	109.5
C10—C9—C8	119.23 (16)	C17—C18—H18C	109.5
O3—C10—C11	123.27 (16)	H18A—C18—H18C	109.5
O3—C10—C9	115.32 (15)	H18B—C18—H18C	109.5

C7—N1—N2—C8	171.60 (15)	N2—C8—C9—C14	12.2 (2)
O1—C1—C2—C3	179.24 (15)	N2—C8—C9—C10	−168.70 (15)
C6—C1—C2—C3	−0.6 (2)	C15—O3—C10—C11	5.3 (2)
C1—C2—C3—C4	−0.8 (2)	C15—O3—C10—C9	−176.25 (14)
C2—C3—C4—C5	1.0 (2)	C14—C9—C10—O3	−178.97 (14)
C3—C4—C5—C6	0.2 (2)	C8—C9—C10—O3	1.9 (2)
C4—C5—C6—C1	−1.6 (2)	C14—C9—C10—C11	−0.5 (2)
C4—C5—C6—C7	178.61 (15)	C8—C9—C10—C11	−179.61 (15)
O1—C1—C6—C5	−178.08 (14)	O3—C10—C11—C12	177.63 (14)
C2—C1—C6—C5	1.7 (2)	C9—C10—C11—C12	−0.7 (2)
O1—C1—C6—C7	1.8 (2)	C16—O4—C12—C11	6.8 (2)
C2—C1—C6—C7	−178.44 (14)	C16—O4—C12—C13	−173.16 (15)
N2—N1—C7—O2	−1.3 (2)	C10—C11—C12—O4	−178.49 (15)
N2—N1—C7—C6	−179.38 (13)	C10—C11—C12—C13	1.5 (2)
C5—C6—C7—O2	175.88 (16)	O4—C12—C13—C14	179.01 (14)
C1—C6—C7—O2	−4.0 (2)	C11—C12—C13—C14	−0.9 (3)
C5—C6—C7—N1	−6.1 (2)	C12—C13—C14—C9	−0.3 (3)
C1—C6—C7—N1	174.10 (15)	C10—C9—C14—C13	1.0 (2)
N1—N2—C8—C9	−179.75 (14)	C8—C9—C14—C13	−179.89 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1o···O2	0.86 (1)	1.74 (2)	2.528 (2)	151 (2)
O5—H5o···N2^{i}	0.85 (1)	2.07 (1)	2.847 (2)	152 (2)
N1—H1n···O5	0.86 (1)	2.09 (1)	2.894 (2)	157 (2)

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.