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2-Hydroxy-*N*′-[(1*Z*)-1-(2-hydroxy-5-methylphenyl)-2-methylpropylidene]-benzohydrazide

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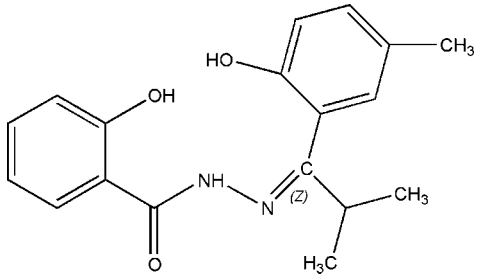
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.042; wR factor = 0.139; data-to-parameter ratio = 13.9.

The title compound, $C_{18}H_{20}N_2O_3$, adopts a *cis* conformation with respect to the C=N double bond. The crystal structure is stabilized by intramolecular N—H···O and intermolecular O—H···O hydrogen bonds.

Related literature

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



Experimental

Crystal data

$C_{18}H_{20}N_2O_3$
M_r = 312.36
Monoclinic, $P2_1/c$
a = 11.2144 (11) Å
b = 11.2887 (11) Å
c = 13.6535 (13) Å
β = 107.000 (2)°
V = 1653.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 273 (2) K
0.25 × 0.22 × 0.16 mm

Data collection

Bruker APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.987, T_{max} = 0.991
8523 measured reflections
2924 independent reflections
2094 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.139
S = 1.00
2924 reflections
210 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 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
N1—H1A···O2	0.86	1.98	2.6413 (18)	133
O2—H2···O1^{i}	0.82	1.96	2.7249 (17)	155
O1—H1···O3^{ii}	0.82	1.90	2.6787 (18)	158

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: PK2070).

References

Bruker (2005). *APEX2* (Version 1.27) and *SAINT* (Version 7.12). Bruker AXS Inc., Madison, Wisconsin, USA.
Carcelli, M., Mazza, P., Pelizzi, G. & Zani, F. (1995). *J. Inorg. Biochem.* **57**, 43–62.
Salem, A. A. (1998). *Microchem. J.* **60**, 51–66.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10. Bruker AXS Inc.,Madison, Wisconsin, USA.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.

supporting information

Acta Cryst. (2008). E**64**, o197 [https://doi.org/10.1107/S160053680706357X]

2-Hydroxy-*N*′-[(1*Z*)-1-(2-hydroxy-5-methylphenyl)-2-methylpropylidene]benzohydrazide

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S1. Comment

The chemistry of aroylhydrazones continues to attract much attention due to their ability to coordinate metal ions (Salem, 1998) and their biological activity (Carcelli *et al.*, 1995). 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 *cis* conformation with respect to the C8=N2 double bond (Fig. 1). The dihedral angle between the two benzene rings is 75.01 (6)°. The crystal structure is stabilized by intramolecular N—H···O and intermolecular O—H···O hydrogen bonds (Table 1. and Fig. 2).

S2. Experimental

2-hydroxybenzohydrazide (0.01 mol,1.52 g) was dissolved in anhydrous ethanol (50 ml) and 1-(2-hydroxy-5-methylphenyl)-2-methylpropan-1-one (0.01 mol, 1.78 g) was added. The reaction mixture was refluxed for 6 h with stirring, and the resulting precipitate was collected by filtration, washed several times with ethanol and dried *in vacuo* (yield 85%). The compound (1.0 mmol, 0.31 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(tertiary) = 0.98 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}($C_{aromatic}$, $C_{tertiary}$, N).

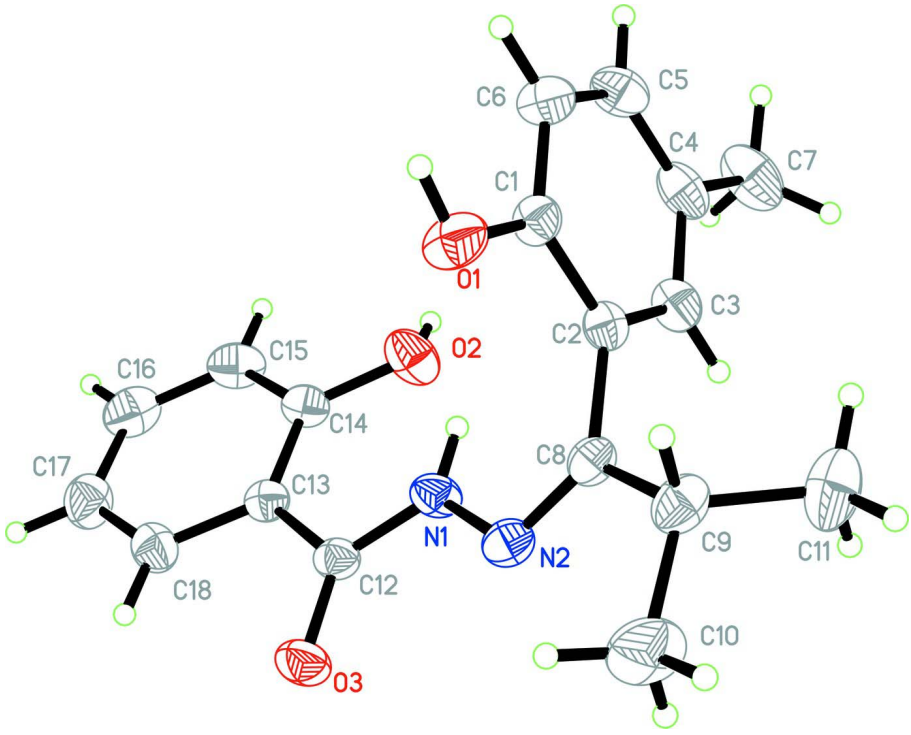

Figure 1

The molecular structure of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

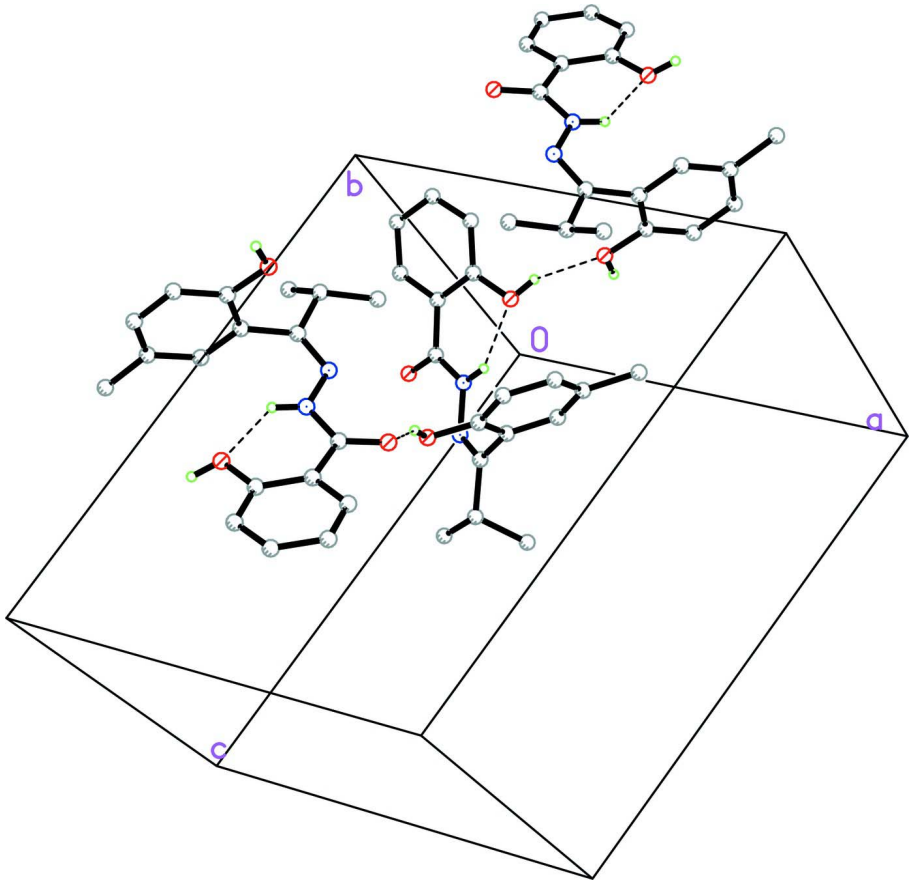

Figure 2

A packing diagram showing intramolecular N—H···O and intermolecular O—H···O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted for clarity.

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Crystal data

$C_{18}H_{20}N_2O_3$
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Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.2144\ (11)$ Å
$b = 11.2887\ (11)$ Å
$c = 13.6535\ (13)$ Å
$\beta = 107.000\ (2)°$
$V = 1653.0\ (3)$ Å^3
$Z = 4$
$F(000) = 664$
$D_x = 1.255$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2019 reflections
$\theta = 2.6–23.2°$
$\mu = 0.09$ mm^{-1}
$T = 273$ K
Block, yellow
0.25 × 0.22 × 0.16 mm

Data collection

Bruker APEX2 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.987$, $T_{max} = 0.991$
8523 measured reflections
2924 independent reflections
2094 reflections with $I > 2\sigma(I)$

R_{int} = 0.026
θ_{max} = 25.1°, θ_{min} = 1.9°
h = −13→10
k = −12→13
l = −10→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.139
S = 1.00
2924 reflections
210 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
$w = 1/[\sigma^2(F_o^2) + (0.0879P)^2]$ 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}

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^ > 2σ(*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	0.14313 (13)	0.32510 (11)	0.31013 (11)	0.0609 (4)
H1	0.1500	0.3965	0.3215	0.091*
O2	0.01561 (11)	0.20180 (13)	−0.04931 (10)	0.0629 (4)
H2	0.0461	0.2149	−0.0960	0.094*
O3	−0.20731 (11)	0.03822 (11)	0.10448 (9)	0.0555 (4)
N1	−0.01360 (13)	0.10594 (12)	0.11865 (10)	0.0445 (4)
H1A	0.0346	0.1442	0.0910	0.053*
N2	0.03470 (14)	0.05414 (12)	0.21453 (11)	0.0444 (4)
C1	0.21322 (16)	0.29390 (14)	0.24792 (13)	0.0426 (4)
C2	0.21798 (15)	0.17485 (15)	0.22370 (12)	0.0397 (4)
C3	0.28605 (16)	0.14187 (17)	0.15787 (14)	0.0496 (5)
H3	0.2892	0.0623	0.1412	0.060*
C4	0.34956 (16)	0.22417 (19)	0.11622 (14)	0.0546 (5)
C5	0.34352 (18)	0.34066 (19)	0.14234 (14)	0.0566 (5)
H5	0.3857	0.3972	0.1156	0.068*
C6	0.27685 (18)	0.37622 (17)	0.20703 (14)	0.0553 (5)
H6	0.2744	0.4560	0.2235	0.066*
C7	0.4215 (2)	0.1856 (2)	0.04319 (17)	0.0788 (7)
H7A	0.4735	0.2495	0.0337	0.118*
H7B	0.3640	0.1644	−0.0216	0.118*
H7C	0.4725	0.1184	0.0713	0.118*

C8	0.14484 (16)	0.08723 (14)	0.26466 (13)	0.0416 (4)
C9	0.20075 (18)	0.03997 (16)	0.37216 (14)	0.0538 (5)
H9	0.2179	0.1089	0.4178	0.065*
C10	0.1119 (2)	−0.0383 (2)	0.40763 (17)	0.0856 (8)
H10A	0.0360	0.0042	0.4018	0.128*
H10B	0.1495	−0.0605	0.4777	0.128*
H10C	0.0938	−0.1081	0.3658	0.128*
C11	0.3253 (2)	−0.0202 (2)	0.38364 (17)	0.0832 (7)
H11A	0.3138	−0.0853	0.3366	0.125*
H11B	0.3584	−0.0490	0.4524	0.125*
H11C	0.3824	0.0357	0.3692	0.125*
C12	−0.13535 (15)	0.09610 (14)	0.06947 (13)	0.0399 (4)
C13	−0.18372 (15)	0.16196 (14)	−0.02863 (12)	0.0394 (4)
C14	−0.11049 (17)	0.21255 (15)	−0.08482 (13)	0.0443 (4)
C15	−0.1663 (2)	0.27366 (17)	−0.17470 (15)	0.0580 (5)
H15	−0.1175	0.3059	−0.2125	0.070*
C16	−0.2935 (2)	0.28657 (18)	−0.20782 (16)	0.0655 (6)
H16	−0.3301	0.3283	−0.2678	0.079*
C17	−0.3677 (2)	0.23870 (19)	−0.15361 (16)	0.0633 (6)
H17	−0.4538	0.2483	−0.1762	0.076*
C18	−0.31253 (17)	0.17651 (16)	−0.06546 (15)	0.0522 (5)
H18	−0.3627	0.1431	−0.0293	0.063*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0832 (10)	0.0437 (7)	0.0734 (9)	0.0020 (7)	0.0506 (8)	−0.0056 (7)
O2	0.0526 (8)	0.0929 (11)	0.0496 (8)	−0.0065 (7)	0.0248 (7)	0.0118 (7)
O3	0.0500 (8)	0.0617 (8)	0.0590 (8)	−0.0089 (6)	0.0224 (7)	0.0137 (6)
N1	0.0446 (8)	0.0515 (9)	0.0388 (8)	−0.0079 (6)	0.0144 (7)	0.0083 (7)
N2	0.0531 (9)	0.0425 (8)	0.0390 (8)	−0.0005 (7)	0.0159 (7)	0.0044 (6)
C1	0.0462 (10)	0.0433 (10)	0.0424 (10)	0.0036 (8)	0.0193 (8)	−0.0023 (8)
C2	0.0377 (9)	0.0448 (10)	0.0356 (9)	0.0011 (7)	0.0091 (7)	−0.0026 (7)
C3	0.0451 (10)	0.0563 (11)	0.0489 (11)	−0.0003 (8)	0.0159 (9)	−0.0156 (9)
C4	0.0395 (10)	0.0822 (15)	0.0439 (11)	−0.0058 (9)	0.0150 (9)	−0.0101 (10)
C5	0.0545 (12)	0.0673 (13)	0.0523 (12)	−0.0135 (10)	0.0222 (10)	0.0019 (10)
C6	0.0665 (13)	0.0441 (10)	0.0600 (12)	−0.0056 (9)	0.0258 (10)	−0.0020 (9)
C7	0.0599 (13)	0.119 (2)	0.0680 (15)	−0.0099 (12)	0.0356 (12)	−0.0210 (13)
C8	0.0488 (10)	0.0365 (9)	0.0405 (10)	0.0040 (8)	0.0145 (8)	−0.0015 (8)
C9	0.0671 (13)	0.0478 (11)	0.0421 (11)	0.0050 (9)	0.0090 (9)	0.0029 (8)
C10	0.103 (2)	0.0882 (18)	0.0617 (14)	−0.0075 (14)	0.0178 (14)	0.0322 (13)
C11	0.0838 (17)	0.0863 (17)	0.0682 (15)	0.0305 (13)	0.0047 (13)	0.0085 (13)
C12	0.0441 (10)	0.0372 (9)	0.0424 (10)	−0.0035 (7)	0.0189 (8)	−0.0023 (7)
C13	0.0462 (10)	0.0373 (9)	0.0374 (9)	−0.0018 (7)	0.0161 (8)	−0.0043 (7)
C14	0.0482 (10)	0.0467 (10)	0.0395 (10)	−0.0063 (8)	0.0154 (8)	−0.0023 (8)
C15	0.0756 (15)	0.0597 (12)	0.0397 (11)	−0.0133 (10)	0.0185 (10)	0.0026 (9)
C16	0.0818 (16)	0.0580 (13)	0.0460 (12)	−0.0033 (11)	0.0021 (11)	0.0042 (9)
C17	0.0544 (12)	0.0653 (13)	0.0603 (13)	0.0054 (10)	0.0012 (10)	−0.0001 (11)

C18	0.0468 (11)	0.0572 (11)	0.0542 (12)	−0.0003 (9)	0.0172 (9)	−0.0044 (10)

Geometric parameters (Å, º)

O1—C1	1.3615 (19)	C7—H7C	0.9600
O1—H1	0.8200	C8—C9	1.515 (2)
O2—C14	1.360 (2)	C9—C10	1.513 (3)
O2—H2	0.8200	C9—C11	1.519 (3)
O3—C12	1.2377 (19)	C9—H9	0.9800
N1—C12	1.338 (2)	C10—H10A	0.9600
N1—N2	1.3909 (19)	C10—H10B	0.9600
N1—H1A	0.8600	C10—H10C	0.9600
N2—C8	1.280 (2)	C11—H11A	0.9600
C1—C6	1.385 (2)	C11—H11B	0.9600
C1—C2	1.389 (2)	C11—H11C	0.9600
C2—C3	1.389 (2)	C12—C13	1.489 (2)
C2—C8	1.494 (2)	C13—C18	1.393 (2)
C3—C4	1.389 (3)	C13—C14	1.399 (2)
C3—H3	0.9300	C14—C15	1.387 (3)
C4—C5	1.369 (3)	C15—C16	1.372 (3)
C4—C7	1.518 (2)	C15—H15	0.9300
C5—C6	1.373 (3)	C16—C17	1.376 (3)
C5—H5	0.9300	C16—H16	0.9300
C6—H6	0.9300	C17—C18	1.374 (3)
C7—H7A	0.9600	C17—H17	0.9300
C7—H7B	0.9600	C18—H18	0.9300
C1—O1—H1	109.5	C10—C9—H9	106.6
C14—O2—H2	109.5	C8—C9—H9	106.6
C12—N1—N2	119.78 (13)	C11—C9—H9	106.6
C12—N1—H1A	120.1	C9—C10—H10A	109.5
N2—N1—H1A	120.1	C9—C10—H10B	109.5
C8—N2—N1	115.46 (14)	H10A—C10—H10B	109.5
O1—C1—C6	122.51 (15)	C9—C10—H10C	109.5
O1—C1—C2	117.92 (15)	H10A—C10—H10C	109.5
C6—C1—C2	119.56 (16)	H10B—C10—H10C	109.5
C1—C2—C3	118.68 (16)	C9—C11—H11A	109.5
C1—C2—C8	119.21 (14)	C9—C11—H11B	109.5
C3—C2—C8	122.03 (15)	H11A—C11—H11B	109.5
C2—C3—C4	122.00 (18)	C9—C11—H11C	109.5
C2—C3—H3	119.0	H11A—C11—H11C	109.5
C4—C3—H3	119.0	H11B—C11—H11C	109.5
C5—C4—C3	117.72 (16)	O3—C12—N1	122.33 (15)
C5—C4—C7	121.53 (18)	O3—C12—C13	120.26 (15)
C3—C4—C7	120.74 (19)	N1—C12—C13	117.38 (13)
C4—C5—C6	121.73 (17)	C18—C13—C14	117.82 (16)
C4—C5—H5	119.1	C18—C13—C12	116.76 (14)
C6—C5—H5	119.1	C14—C13—C12	125.40 (15)

C5—C6—C1	120.31 (18)	O2—C14—C15	120.87 (16)
C5—C6—H6	119.8	O2—C14—C13	118.95 (15)
C1—C6—H6	119.8	C15—C14—C13	120.18 (17)
C4—C7—H7A	109.5	C16—C15—C14	120.07 (18)
C4—C7—H7B	109.5	C16—C15—H15	120.0
H7A—C7—H7B	109.5	C14—C15—H15	120.0
C4—C7—H7C	109.5	C15—C16—C17	121.00 (19)
H7A—C7—H7C	109.5	C15—C16—H16	119.5
H7B—C7—H7C	109.5	C17—C16—H16	119.5
N2—C8—C2	122.85 (15)	C18—C17—C16	118.89 (19)
N2—C8—C9	118.46 (15)	C18—C17—H17	120.6
C2—C8—C9	118.62 (15)	C16—C17—H17	120.6
C10—C9—C8	113.01 (17)	C17—C18—C13	122.03 (18)
C10—C9—C11	112.20 (18)	C17—C18—H18	119.0
C8—C9—C11	111.40 (16)	C13—C18—H18	119.0

C12—N1—N2—C8	−166.53 (15)	C2—C8—C9—C10	174.27 (17)
O1—C1—C2—C3	178.11 (16)	N2—C8—C9—C11	124.62 (19)
C6—C1—C2—C3	−0.6 (3)	C2—C8—C9—C11	−58.3 (2)
O1—C1—C2—C8	1.2 (2)	N2—N1—C12—O3	−2.8 (2)
C6—C1—C2—C8	−177.50 (16)	N2—N1—C12—C13	174.85 (14)
C1—C2—C3—C4	0.3 (3)	O3—C12—C13—C18	13.7 (2)
C8—C2—C3—C4	177.10 (16)	N1—C12—C13—C18	−163.97 (15)
C2—C3—C4—C5	0.2 (3)	O3—C12—C13—C14	−168.03 (16)
C2—C3—C4—C7	−179.04 (17)	N1—C12—C13—C14	14.3 (2)
C3—C4—C5—C6	−0.3 (3)	C18—C13—C14—O2	178.59 (16)
C7—C4—C5—C6	178.90 (19)	C12—C13—C14—O2	0.4 (2)
C4—C5—C6—C1	0.0 (3)	C18—C13—C14—C15	−0.7 (2)
O1—C1—C6—C5	−178.17 (18)	C12—C13—C14—C15	−178.97 (16)
C2—C1—C6—C5	0.5 (3)	O2—C14—C15—C16	−178.11 (17)
N1—N2—C8—C2	−0.4 (2)	C13—C14—C15—C16	1.2 (3)
N1—N2—C8—C9	176.55 (14)	C14—C15—C16—C17	−0.6 (3)
C1—C2—C8—N2	93.6 (2)	C15—C16—C17—C18	−0.5 (3)
C3—C2—C8—N2	−83.1 (2)	C16—C17—C18—C13	0.9 (3)
C1—C2—C8—C9	−83.3 (2)	C14—C13—C18—C17	−0.3 (3)
C3—C2—C8—C9	99.94 (19)	C12—C13—C18—C17	178.06 (17)
N2—C8—C9—C10	−2.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2	0.86	1.98	2.6413 (18)	133
O2—H2···O1^{i}	0.82	1.96	2.7249 (17)	155
O1—H1···O3^{ii}	0.82	1.90	2.6787 (18)	158

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