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(*E*)-2-(5,5-Dimethylhexahydropyrimidin-2-yl)-4-(phenyldiazenyl)phenol

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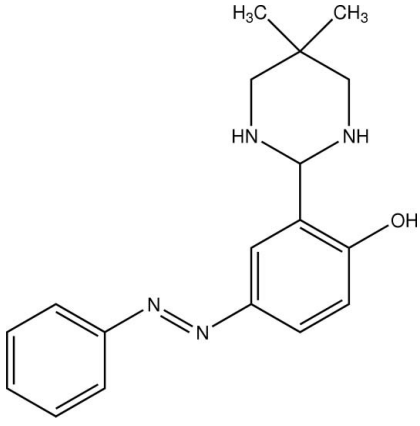
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.003 Å; R factor = 0.050; wR factor = 0.114; data-to-parameter ratio = 13.4.

In the title Schiff base, $C_{18}H_{22}N_4O$, the hexahydropyrimidinyl ring adopts a chair conformation. The dihedral angle between the aromatic rings of the 4-(2-phenyldiazenyl)phenol unit is 15.7 (1)°. There is an intramolecular O—H···N hydrogen bond between the hydroxyl group and an N atom of the hexahydropyimidinyl unit. Intermolecular N—H···O and N—H···N hydrogen bonds give rise to a layer structure.

Related literature

For applications and related structures, see: Farrell *et al.* (2007).



Experimental

Crystal data

$C_{18}H_{22}N_4O$
M_r = 310.40
Orthorhombic, *Pbca*
a = 9.0287 (9) Å
b = 12.0767 (12) Å
c = 30.866 (3) Å
V = 3365.5 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 120 (2) K
0.23 × 0.20 × 0.16 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.970, T_{max} = 0.987
14483 measured reflections
3134 independent reflections
1574 reflections with $I > 2\sigma(I)$
R_{int} = 0.078

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.114
S = 0.81
3134 reflections
234 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N3	1.01 (2)	1.65 (2)	2.584 (2)	152 (2)
N3—H3A···N4^{i}	0.92 (2)	2.30 (2)	3.159 (3)	155 (2)
N4—H4A···O1^{ii}	0.93 (2)	2.18 (2)	3.106 (3)	172 (2)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2518).

References

Brandenburg, K. (2001). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (1998). *SAINT-Plus* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrell, J. R., Niconchuk, J., Higham, C. S. & Bergeron, B. W. (2007). *Tetrahedron Lett.* **48**, 8034–8036.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supporting information

Acta Cryst. (2008). E**64**, o2453 [doi:10.1107/S1600536808038877]

(*E*)-2-(5,5-Dimethylhexahydropyrimidin-2-yl)-4-(phenyldiazenyl)phenol

Iran Sheikhshoaie, Niaz Monadi and Alireza Abbasi

S1. Comment

Heterocycles containing nitrogen atoms *e.g.* hexahydropyrimidines have applications in both inorganic and organic chemistry. Hexahydropyrimidines can be easily prepared from condensations of alkyl diamines and aldehydes. Our interest in synthesizing derivatives of these heterocycles was due to their anti-carcinoma, anti-lymphoma, and anti-biotic properties.

The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. Two aromatic rings A (C5—C10) and B (C11—C16) show a little deviation from planarity with a dihedral angle of 15.7 (1)^{o}. Hexahydropyrimidine has a chair conformation. Intramolecular hydrogen bonds are formed between the phenol hydroxyl groups and the nearest N atom in the hexahydropyimidine groups [O—H···N = 2.584 (2) Å]. The packing of the structure is stabilized by relatively strong N—H···O & N—H···N hydrogen bonds (see Tab. 1), and C—H···π contacts [C—H-cetroid = 2.70 Å] between neighboring molecules. No significant π-π interactions are found in the crystal structure.

S2. Experimental

The title compound was prepared *via* condensation of (*E*)-5-(2-phenyldiazenyl)-2-hydroxybenzaldehyde and 2,2-dimethylpropane-1,3-diamine in 20 ml EtOH:CHCl3. The mixture solution was stirred and refluxed for 3 h. Colorless prismatic-shape crystals were obtained after evaporation of the excess solvent.

S3. Refinement

Aromatic and methyl H atoms were placed in calculated positions (C—H = 0.93 Å, C—H = 0.96 Å, respectively) and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 and 1.5 U_{eq}(C), respectively. Methylene, hydroxyl and amine H atoms were located in difference density maps and their coordinates were refined freely with U_{iso}(H) = 1.5 U_{eq}(C, O & N).

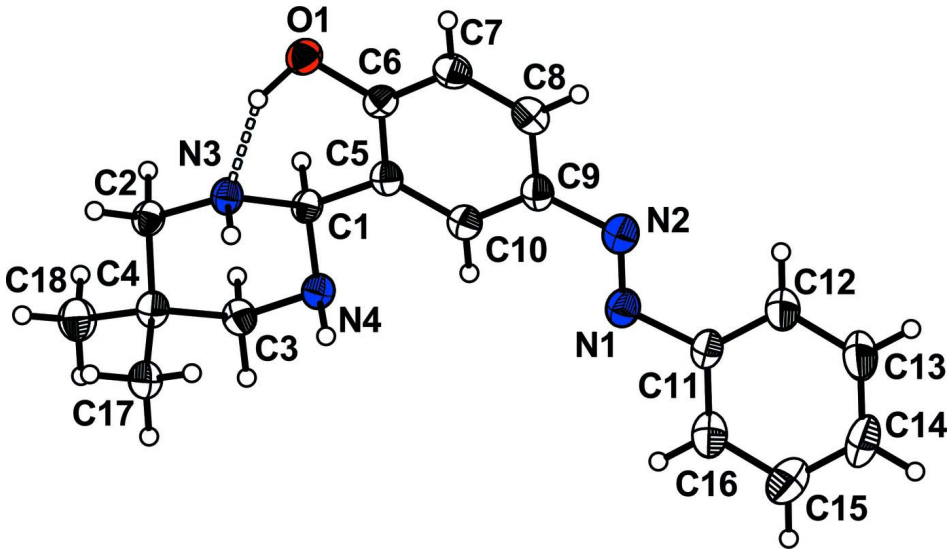

Figure 1

Molecular structure of (I), with 50% probability displacement ellipsoids. H atoms are shown as circles of arbitrary radii.

(*E*)-2-(5,5-Dimethylhexahydropyrimidin-2-yl)-4-(phenyldiazenyl)phenol

Crystal data

$C_{18}H_{22}N_4O$
$M_r = 310.40$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 9.0287\ (9)$ Å
$b = 12.0767\ (12)$ Å
$c = 30.866\ (3)$ Å
$V = 3365.5\ (6)$ Å^3
$Z = 8$
$F(000) = 1328$
$D_x = 1.225$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 14483 reflections
$\theta = 3$–60°
$\mu = 0.08$ mm^{-1}
$T = 120$ K
Prism, colorless
0.23 × 0.20 × 0.16 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.970$, $T_{max} = 0.987$
14483 measured reflections
3134 independent reflections
1574 reflections with $I > 2\sigma(I)$
$R_{int} = 0.078$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.6°$
$h = -10 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -32 \rightarrow 37$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.114$
$S = 0.81$
3134 reflections
234 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

$w = 1/[\sigma^2(F_o^2) + (0.049P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.69820 (17)	0.17061 (13)	0.26940 (5)	0.0314 (4)
H1A	0.710 (3)	0.121 (2)	0.2433 (7)	0.047*
N1	0.9722 (2)	−0.15729 (17)	0.40388 (6)	0.0301 (5)
N2	0.9051 (2)	−0.06703 (18)	0.41075 (6)	0.0312 (5)
N3	0.7588 (2)	0.00338 (17)	0.22029 (6)	0.0259 (5)
H3A	0.692 (3)	−0.0499 (19)	0.2283 (7)	0.039*
N4	0.9679 (2)	−0.11263 (17)	0.24545 (6)	0.0260 (5)
H4A	0.911 (3)	−0.1732 (19)	0.2543 (7)	0.039*
C1	0.8894 (3)	−0.0082 (2)	0.24903 (7)	0.0237 (6)
H1	0.964 (2)	0.0526 (18)	0.2397 (6)	0.036*
C2	0.8017 (3)	−0.0160 (2)	0.17456 (7)	0.0261 (6)
H21	0.876 (3)	0.0485 (19)	0.1681 (7)	0.039*
H22	0.707 (2)	−0.0077 (18)	0.1561 (7)	0.039*
C3	1.0059 (3)	−0.1371 (2)	0.20003 (8)	0.0277 (6)
H31	1.087 (2)	−0.081 (2)	0.1916 (7)	0.042*
H32	1.048 (2)	−0.212 (2)	0.1986 (7)	0.042*
C4	0.8774 (2)	−0.1275 (2)	0.16755 (7)	0.0265 (6)
C5	0.8460 (2)	0.0185 (2)	0.29537 (7)	0.0237 (6)
C6	0.7534 (2)	0.11069 (19)	0.30298 (7)	0.0244 (6)
C7	0.7169 (3)	0.1417 (2)	0.34482 (7)	0.0306 (6)
H7	0.6563	0.2028	0.3495	0.037*
C8	0.7703 (3)	0.0821 (2)	0.37939 (7)	0.0302 (6)
H8	0.7471	0.1039	0.4075	0.036*
C9	0.8586 (2)	−0.0104 (2)	0.37271 (7)	0.0267 (6)
C10	0.8971 (2)	−0.0407 (2)	0.33038 (7)	0.0261 (6)
H10	0.9581	−0.1017	0.3259	0.031*
C11	1.0190 (3)	−0.2133 (2)	0.44232 (7)	0.0295 (6)
C12	1.0096 (3)	−0.1697 (2)	0.48356 (7)	0.0423 (7)
H12	0.9718	−0.0989	0.4878	0.051*
C13	1.0566 (3)	−0.2317 (3)	0.51851 (8)	0.0508 (8)
H13	1.0503	−0.2024	0.5463	0.061*
C14	1.1127 (3)	−0.3367 (3)	0.51259 (8)	0.0501 (8)

H14	1.1445	−0.3778	0.5363	0.060*
C15	1.1217 (3)	−0.3807 (3)	0.47163 (8)	0.0481 (8)
H15	1.1587	−0.4518	0.4676	0.058*
C16	1.0754 (3)	−0.3187 (2)	0.43638 (8)	0.0404 (7)
H16	1.0822	−0.3481	0.4086	0.049*
C17	0.7683 (3)	−0.22207 (19)	0.17308 (7)	0.0326 (6)
H17A	0.7266	−0.2195	0.2017	0.049*
H17B	0.8186	−0.2913	0.1690	0.049*
H17C	0.6905	−0.2152	0.1520	0.049*
C18	0.9416 (3)	−0.1313 (2)	0.12165 (7)	0.0371 (7)
H18A	0.8627	−0.1258	0.1009	0.056*
H18B	0.9936	−0.1998	0.1175	0.056*
H18C	1.0089	−0.0705	0.1177	0.056*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0319 (10)	0.0309 (11)	0.0312 (10)	0.0044 (8)	0.0002 (8)	0.0017 (8)
N1	0.0269 (12)	0.0339 (13)	0.0294 (12)	−0.0031 (11)	−0.0025 (9)	0.0034 (10)
N2	0.0262 (12)	0.0355 (13)	0.0320 (12)	−0.0021 (11)	−0.0005 (10)	0.0029 (10)
N3	0.0220 (11)	0.0308 (13)	0.0249 (11)	−0.0014 (10)	−0.0025 (9)	0.0016 (10)
N4	0.0241 (12)	0.0288 (12)	0.0250 (11)	−0.0012 (10)	0.0007 (9)	0.0020 (10)
C1	0.0190 (13)	0.0262 (14)	0.0259 (13)	0.0022 (12)	−0.0040 (11)	0.0008 (11)
C2	0.0242 (14)	0.0292 (15)	0.0250 (14)	0.0024 (12)	0.0000 (11)	0.0011 (11)
C3	0.0239 (14)	0.0315 (16)	0.0277 (14)	0.0011 (12)	−0.0007 (11)	−0.0021 (12)
C4	0.0236 (13)	0.0310 (15)	0.0250 (13)	−0.0040 (12)	−0.0002 (11)	0.0021 (11)
C5	0.0173 (12)	0.0276 (14)	0.0260 (13)	−0.0033 (11)	−0.0014 (10)	−0.0003 (11)
C6	0.0205 (13)	0.0237 (14)	0.0290 (13)	−0.0048 (11)	0.0002 (12)	0.0031 (11)
C7	0.0286 (15)	0.0290 (15)	0.0342 (15)	0.0004 (12)	0.0018 (12)	−0.0032 (12)
C8	0.0282 (15)	0.0328 (15)	0.0297 (14)	−0.0045 (13)	0.0040 (12)	−0.0040 (11)
C9	0.0241 (14)	0.0308 (15)	0.0251 (13)	−0.0072 (12)	−0.0008 (11)	0.0014 (11)
C10	0.0203 (13)	0.0284 (15)	0.0295 (14)	−0.0005 (11)	−0.0008 (11)	−0.0008 (11)
C11	0.0282 (14)	0.0354 (16)	0.0250 (14)	−0.0024 (13)	−0.0027 (11)	0.0050 (12)
C12	0.0562 (19)	0.0416 (18)	0.0292 (15)	−0.0041 (15)	−0.0075 (14)	0.0021 (14)
C13	0.066 (2)	0.058 (2)	0.0279 (15)	−0.0043 (18)	−0.0083 (14)	0.0001 (15)
C14	0.055 (2)	0.063 (2)	0.0325 (17)	−0.0007 (17)	−0.0086 (15)	0.0163 (15)
C15	0.0481 (19)	0.050 (2)	0.0462 (18)	0.0091 (16)	−0.0025 (15)	0.0125 (15)
C16	0.0401 (17)	0.0511 (19)	0.0300 (15)	0.0032 (15)	−0.0031 (13)	0.0007 (13)
C17	0.0339 (15)	0.0339 (16)	0.0299 (13)	0.0007 (13)	−0.0056 (12)	−0.0002 (11)
C18	0.0368 (16)	0.0435 (17)	0.0310 (14)	0.0005 (14)	0.0038 (12)	−0.0007 (13)

Geometric parameters (Å, º)

O1—C6	1.358 (2)	C7—C8	1.375 (3)
O1—H1A	1.01 (2)	C7—H7	0.9300
N1—N2	1.265 (3)	C8—C9	1.387 (3)
N1—C11	1.429 (3)	C8—H8	0.9300
N2—C9	1.422 (3)	C9—C10	1.401 (3)

N3—C2	1.482 (3)	C10—H10	0.9300
N3—C1	1.483 (3)	C11—C12	1.380 (3)
N3—H3A	0.92 (2)	C11—C16	1.384 (3)
N4—C1	1.451 (3)	C12—C13	1.380 (3)
N4—C3	1.473 (3)	C12—H12	0.9300
N4—H4A	0.93 (2)	C13—C14	1.377 (4)
C1—C5	1.518 (3)	C13—H13	0.9300
C1—H1	1.04 (2)	C14—C15	1.374 (3)
C2—C4	1.525 (3)	C14—H14	0.9300
C2—H21	1.05 (2)	C15—C16	1.385 (3)
C2—H22	1.03 (2)	C15—H15	0.9300
C3—C4	1.537 (3)	C16—H16	0.9300
C3—H31	1.03 (2)	C17—H17A	0.9600
C3—H32	0.98 (2)	C17—H17B	0.9600
C4—C17	1.518 (3)	C17—H17C	0.9600
C4—C18	1.531 (3)	C18—H18A	0.9600
C5—C10	1.375 (3)	C18—H18B	0.9600
C5—C6	1.412 (3)	C18—H18C	0.9600
C6—C7	1.385 (3)		

C6—O1—H1A	104.5 (13)	C6—C7—H7	120.1
N2—N1—C11	114.21 (19)	C7—C8—C9	120.5 (2)
N1—N2—C9	114.67 (19)	C7—C8—H8	119.7
C2—N3—C1	110.27 (18)	C9—C8—H8	119.7
C2—N3—H3A	108.7 (14)	C8—C9—C10	119.4 (2)
C1—N3—H3A	107.3 (14)	C8—C9—N2	115.7 (2)
C1—N4—C3	111.13 (19)	C10—C9—N2	124.8 (2)
C1—N4—H4A	113.1 (14)	C5—C10—C9	120.9 (2)
C3—N4—H4A	104.4 (14)	C5—C10—H10	119.5
N4—C1—N3	115.15 (19)	C9—C10—H10	119.5
N4—C1—C5	112.51 (19)	C12—C11—C16	119.7 (2)
N3—C1—C5	109.76 (18)	C12—C11—N1	124.5 (2)
N4—C1—H1	106.2 (12)	C16—C11—N1	115.7 (2)
N3—C1—H1	106.5 (12)	C11—C12—C13	119.6 (3)
C5—C1—H1	106.1 (11)	C11—C12—H12	120.2
N3—C2—C4	113.06 (19)	C13—C12—H12	120.2
N3—C2—H21	103.4 (12)	C14—C13—C12	120.6 (2)
C4—C2—H21	110.0 (13)	C14—C13—H13	119.7
N3—C2—H22	107.0 (12)	C12—C13—H13	119.7
C4—C2—H22	112.2 (13)	C15—C14—C13	120.0 (3)
H21—C2—H22	110.8 (17)	C15—C14—H14	120.0
N4—C3—C4	115.46 (19)	C13—C14—H14	120.0
N4—C3—H31	105.8 (12)	C14—C15—C16	119.8 (3)
C4—C3—H31	108.8 (12)	C14—C15—H15	120.1
N4—C3—H32	108.5 (13)	C16—C15—H15	120.1
C4—C3—H32	109.2 (13)	C11—C16—C15	120.3 (2)
H31—C3—H32	108.9 (18)	C11—C16—H16	119.9
C17—C4—C2	110.9 (2)	C15—C16—H16	119.9

C17—C4—C18	109.10 (19)	C4—C17—H17A	109.5
C2—C4—C18	109.10 (19)	C4—C17—H17B	109.5
C17—C4—C3	111.07 (19)	H17A—C17—H17B	109.5
C2—C4—C3	108.18 (19)	C4—C17—H17C	109.5
C18—C4—C3	108.39 (19)	H17A—C17—H17C	109.5
C10—C5—C6	118.6 (2)	H17B—C17—H17C	109.5
C10—C5—C1	122.9 (2)	C4—C18—H18A	109.5
C6—C5—C1	118.5 (2)	C4—C18—H18B	109.5
O1—C6—C7	118.7 (2)	H18A—C18—H18B	109.5
O1—C6—C5	120.7 (2)	C4—C18—H18C	109.5
C7—C6—C5	120.6 (2)	H18A—C18—H18C	109.5
C8—C7—C6	119.9 (2)	H18B—C18—H18C	109.5
C8—C7—H7	120.1		

C11—N1—N2—C9	179.73 (19)	O1—C6—C7—C8	179.6 (2)
C3—N4—C1—N3	52.1 (3)	C5—C6—C7—C8	−0.4 (3)
C3—N4—C1—C5	178.90 (19)	C6—C7—C8—C9	−1.2 (4)
C2—N3—C1—N4	−55.2 (3)	C7—C8—C9—C10	2.1 (3)
C2—N3—C1—C5	176.64 (19)	C7—C8—C9—N2	−178.7 (2)
C1—N3—C2—C4	55.4 (3)	N1—N2—C9—C8	173.0 (2)
C1—N4—C3—C4	−50.2 (3)	N1—N2—C9—C10	−7.9 (3)
N3—C2—C4—C17	69.7 (2)	C6—C5—C10—C9	−0.1 (3)
N3—C2—C4—C18	−170.09 (19)	C1—C5—C10—C9	177.5 (2)
N3—C2—C4—C3	−52.4 (3)	C8—C9—C10—C5	−1.4 (3)
N4—C3—C4—C17	−71.9 (3)	N2—C9—C10—C5	179.4 (2)
N4—C3—C4—C2	50.1 (3)	N2—N1—C11—C12	−7.3 (3)
N4—C3—C4—C18	168.3 (2)	N2—N1—C11—C16	171.9 (2)
N4—C1—C5—C10	10.1 (3)	C16—C11—C12—C13	0.0 (4)
N3—C1—C5—C10	139.7 (2)	N1—C11—C12—C13	179.2 (2)
N4—C1—C5—C6	−172.34 (19)	C11—C12—C13—C14	0.0 (4)
N3—C1—C5—C6	−42.7 (3)	C12—C13—C14—C15	−0.3 (4)
C10—C5—C6—O1	−178.9 (2)	C13—C14—C15—C16	0.6 (4)
C1—C5—C6—O1	3.4 (3)	C12—C11—C16—C15	0.3 (4)
C10—C5—C6—C7	1.0 (3)	N1—C11—C16—C15	−179.0 (2)
C1—C5—C6—C7	−176.7 (2)	C14—C15—C16—C11	−0.6 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N3	1.01 (2)	1.65 (2)	2.584 (2)	152 (2)
N3—H3*A*···N4^{i}	0.92 (2)	2.30 (2)	3.159 (3)	155 (2)
N4—H4*A*···O1^{ii}	0.93 (2)	2.18 (2)	3.106 (3)	172 (2)

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