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ISSN 2414-3146

(4*E*)-1-Phenyl-4-[(piperidin-1-yl)methylidene]-pyrazolidine-3,5-dione

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Received 19 June 2016
Accepted 24 June 2016

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

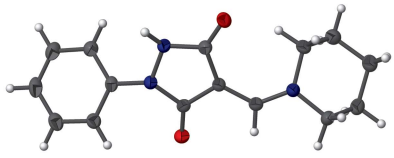
Keywords: crystal structure; pyrazolidindiones; β-unsaturated carbonyl compounds.

CCDC reference: 1487642

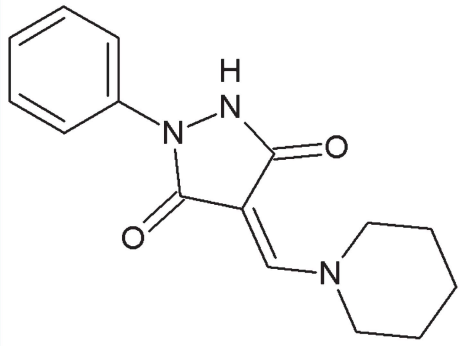
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{15}H_{17}N_3O_2$, the dihedral angle between the planes of the pyrazolidine and phenyl rings is 29.91 (6)°. The piperidine ring adopts a chair conformation. In the crystal, molecules are linked into chains running parallel to the *a*-axis direction by a combination of N—H···O and C—H···O hydrogen bonds. Furthermore, there exist C—H···π interactions and π–π stacking interactions [centroid-to-centroid distance = 3.5274 (10) Å] between the pyrazolidine rings of adjacent molecules.

3D view



Chemical scheme



Structure description

Pyrazolone derivatives have diverse pharmacological properties, such as cytotoxic, anti-inflammatory, antimicrobial, antioxidant, antifungal, antiviral and oral hypoglycaemic activity (Kumar *et al.*, 2012). As part of our studies in this area, the synthesis and structure of the title compound are reported.

In the title compound, the piperidine ring (atoms N3/C5–C9) adopts a chair conformation, with the puckering parameters Q_T = 0.547 (2) Å, θ = 176.2 (2)° and φ = 344 (3)°. The dihedral angle between the planes of the phenyl and pyrazolidine rings is 29.91 (6)°. The molecular conformation is partially determined by an intramolecular C5—H5*B*···O1 hydrogen bond (Table 1 and Fig. 1).

In the crystal, molecules pack in columns running parallel to the *a* axis which are assembled by N2—H2···O1^{i} and a combination of C9—H9*A*···O1^{ii} and C11—H11···O2^{iii} hydrogen bonds [symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $x-1, y, z$;

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

*Cg*3 is the centroid of the C10–C15 phenyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.94 (2)	1.86 (2)	2.8008 (18)	171 (2)
C5—H5*B*···O1	0.99	2.24	2.979 (2)	131
C9—H9*A*···O1^{ii}	0.99	2.51	3.421 (2)	152
C11—H11···O2^{iii}	0.95	2.52	3.471 (2)	178
C9—H9*B*···*Cg*3^{iv}	0.99	2.91	3.741 (2)	143

Symmetry codes: (i) $-x+2, -y+1, -z+1$; (ii) $x-1, y, z$; (iii) $x+1, y, z$; (iv) $-x+1, -y+1, -z+1$.

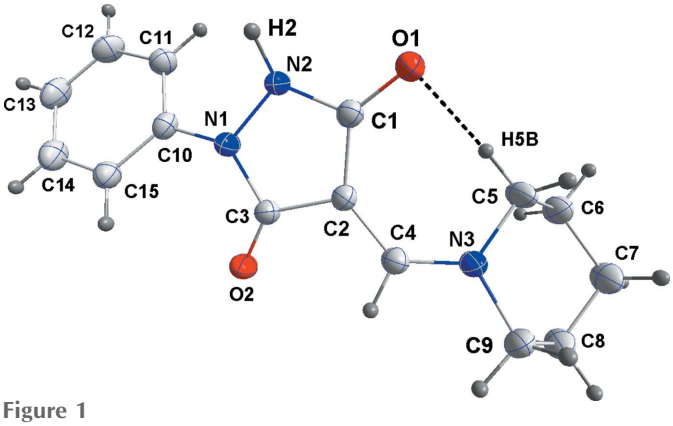

Figure 1
The title molecule, shown with 50% probability displacement ellipsoids. The intramolecular C—H···O hydrogen bond is shown by a dotted line.

(iii) $x+1, y, z$] (Table 1 and Figs. 2 and 3). In addition, C—H···π interactions (Table 1) and π–π stacking interactions [*Cg*1···*Cg*1($-x+1, -y+1, -z+1$) = 3.5274 (10) Å; where *Cg*1 is the centroid of the pyrazolidine ring (N1/N2/C1–C3)] are observed in the crystal structure.

Synthesis and crystallization

A mixture of 1 mmol (231.3 mg) of 4-[(dimethylamino)-methylidene]-1-phenylpyrazolidine-3,5-dione and 1 mmol (85 mg) of piperidine was refluxed in 30 ml dioxane for 2 h. The obtained solid product was collected under vacuum and recrystallized from ethanol to yield colourless rods of the title compound (yield 95%; m.p. 518–520 K).

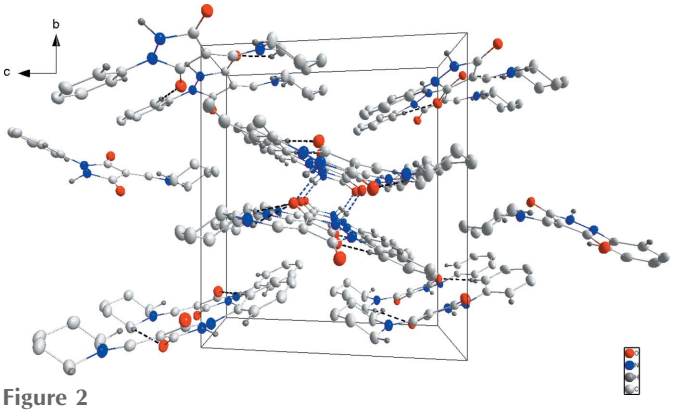

Figure 2
The packing, viewed along the *a* axis, with intermolecular N—H···O and C—H···O hydrogen bonds shown, respectively, as blue and black dotted lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{17}N_3O_2$
M_r	271.32
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, *b*, *c* (Å)	6.3184 (2), 15.4673 (6), 13.2528 (5)
β (°)	98.762 (2)
V (Å^3)	1280.06 (8)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.78
Crystal size (mm)	0.12 × 0.07 × 0.04
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*TWINABS*; Sheldrick, 2009)
T_{min}, T_{max}	0.81, 0.97
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	16143, 2493, 1946
R_{int}	0.053
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.043, 0.109, 1.03
No. of reflections	2493
No. of parameters	186
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.21

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Bruker, 2016).

Refinement

Trial refinements with both the single-component reflection file extracted from the full data set with *TWINABS* (Sheldrick, 2009) and with the complete two-component reflection file indicated the former to provide superior results. Crystal data, data collection and structure refinement details are summarized in Table 2.

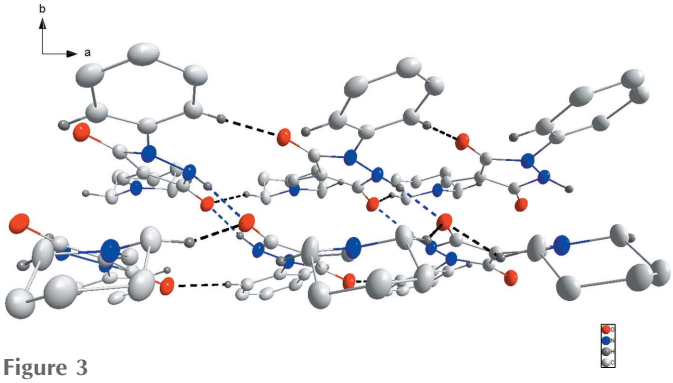

Figure 3
Side view of a portion of the central chain shown in Fig. 2, with intermolecular N—H···O and C—H···O hydrogen bonds shown, respectively, as blue and black dotted lines.

Acknowledgements

The support of NSF–MRI grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged.

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full crystallographic data

IUCrData (2016). **1**, x161028 [doi:10.1107/S2414314616010282]

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Crystal data

$C_{15}H_{17}N_3O_2$
M_r = 271.32
Monoclinic, $P2_1/c$
a = 6.3184 (2) Å
b = 15.4673 (6) Å
c = 13.2528 (5) Å
β = 98.762 (2)°
V = 1280.06 (8) Å^3
Z = 4
$F(000)$ = 576
D_x = 1.408 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 6240 reflections
θ = 4.4–72.2°
μ = 0.78 mm^{-1}
T = 150 K
Rod, colourless
0.12 × 0.07 × 0.04 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*TWINABS*; Sheldrick, 2009)
T_{min} = 0.81, T_{max} = 0.97
16143 measured reflections
2493 independent reflections
1946 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 72.5°, θ_{min} = 4.4°
h = −7→7
k = −18→18
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.109
S = 1.03
2493 reflections
186 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.3794P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015*b*), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0047 (6)

Special details

Experimental. Analysis of 961 reflections having I/σ(I) > 12 and chosen from the full data set with *CELL_NOW* (Sheldrick, 2008) showed the crystal to belong to the monoclinic system and to be twinned by a 180° rotation about the *a* axis. The raw data were processed using the multi-component version of*SAINT* under control of the two-component orientation file generated by *CELL_NOW*.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\mathrm{sigma}(F^2)$ 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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.99 Å) and included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.84360 (19)	0.52001 (8)	0.37140 (9)	0.0280 (3)
O2	0.31975 (19)	0.66186 (8)	0.53428 (9)	0.0305 (3)
N1	0.6720 (2)	0.61623 (9)	0.57952 (10)	0.0246 (3)
N2	0.8306 (2)	0.58417 (9)	0.52548 (10)	0.0239 (3)
H2	0.943 (4)	0.5538 (15)	0.5650 (16)	0.041 (6)*
N3	0.3412 (2)	0.56277 (10)	0.24051 (10)	0.0273 (3)
C1	0.7405 (3)	0.56111 (10)	0.42895 (12)	0.0230 (4)
C2	0.5203 (3)	0.59034 (11)	0.41576 (12)	0.0225 (4)
C3	0.4811 (3)	0.62622 (11)	0.51260 (12)	0.0231 (4)
C4	0.3478 (3)	0.58062 (11)	0.33819 (12)	0.0245 (4)
H4	0.2116	0.5881	0.3590	0.029*
C5	0.5288 (3)	0.56342 (15)	0.18738 (13)	0.0355 (5)
H5A	0.5579	0.5040	0.1653	0.043*
H5B	0.6558	0.5836	0.2346	0.043*
C6	0.4900 (3)	0.62225 (14)	0.09547 (14)	0.0346 (4)
H6A	0.6133	0.6186	0.0577	0.042*
H6B	0.4789	0.6827	0.1185	0.042*
C7	0.2870 (3)	0.59810 (14)	0.02483 (14)	0.0362 (5)
H7A	0.3024	0.5396	−0.0036	0.043*
H7B	0.2619	0.6395	−0.0327	0.043*
C8	0.0982 (3)	0.59946 (14)	0.08315 (14)	0.0354 (5)
H8A	0.0734	0.6594	0.1047	0.042*
H8B	−0.0320	0.5799	0.0378	0.042*
C9	0.1371 (3)	0.54169 (13)	0.17623 (13)	0.0314 (4)
H9A	0.0185	0.5488	0.2165	0.038*
H9B	0.1392	0.4806	0.1543	0.038*
C10	0.7371 (3)	0.65691 (11)	0.67417 (12)	0.0239 (4)
C11	0.9487 (3)	0.68439 (11)	0.70058 (13)	0.0280 (4)
H11	1.0486	0.6769	0.6545	0.034*
C12	1.0114 (3)	0.72278 (12)	0.79519 (14)	0.0336 (4)

H12	1.1551	0.7417	0.8136	0.040*
C13	0.8670 (3)	0.73380 (12)	0.86315 (14)	0.0357 (5)
H13	0.9109	0.7605	0.9275	0.043*
C14	0.6583 (3)	0.70556 (12)	0.83624 (14)	0.0332 (4)
H14	0.5591	0.7131	0.8827	0.040*
C15	0.5910 (3)	0.66642 (11)	0.74251 (13)	0.0277 (4)
H15	0.4479	0.6465	0.7252	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0264 (6)	0.0348 (7)	0.0231 (6)	0.0078 (5)	0.0045 (5)	−0.0008 (5)
O2	0.0232 (6)	0.0392 (7)	0.0293 (7)	0.0055 (5)	0.0044 (5)	−0.0031 (5)
N1	0.0206 (7)	0.0311 (8)	0.0220 (7)	0.0034 (6)	0.0028 (6)	−0.0030 (6)
N2	0.0189 (7)	0.0306 (8)	0.0216 (7)	0.0049 (6)	0.0014 (6)	−0.0017 (6)
N3	0.0210 (7)	0.0391 (9)	0.0215 (7)	0.0018 (6)	0.0025 (6)	0.0006 (6)
C1	0.0229 (8)	0.0239 (8)	0.0222 (8)	0.0005 (6)	0.0029 (7)	0.0017 (6)
C2	0.0218 (8)	0.0246 (8)	0.0209 (8)	0.0010 (6)	0.0028 (6)	0.0011 (6)
C3	0.0204 (8)	0.0252 (8)	0.0234 (8)	−0.0002 (6)	0.0027 (6)	0.0011 (6)
C4	0.0225 (8)	0.0272 (8)	0.0241 (8)	0.0011 (6)	0.0042 (7)	0.0021 (6)
C5	0.0226 (9)	0.0612 (13)	0.0233 (9)	0.0078 (8)	0.0054 (7)	0.0039 (8)
C6	0.0312 (10)	0.0435 (11)	0.0297 (10)	−0.0031 (8)	0.0063 (8)	0.0024 (8)
C7	0.0343 (10)	0.0466 (12)	0.0270 (10)	0.0050 (8)	0.0028 (8)	0.0054 (8)
C8	0.0276 (10)	0.0491 (12)	0.0277 (10)	0.0065 (8)	−0.0012 (8)	−0.0009 (8)
C9	0.0230 (9)	0.0449 (11)	0.0255 (9)	−0.0037 (7)	0.0012 (7)	−0.0037 (7)
C10	0.0279 (9)	0.0216 (8)	0.0210 (8)	0.0028 (6)	0.0003 (7)	0.0001 (6)
C11	0.0272 (9)	0.0283 (9)	0.0280 (9)	0.0002 (7)	0.0027 (7)	−0.0010 (7)
C12	0.0332 (10)	0.0303 (10)	0.0344 (10)	0.0000 (7)	−0.0043 (8)	−0.0048 (7)
C13	0.0457 (11)	0.0328 (10)	0.0260 (9)	0.0082 (8)	−0.0032 (8)	−0.0065 (7)
C14	0.0401 (11)	0.0340 (10)	0.0257 (9)	0.0105 (8)	0.0056 (8)	−0.0016 (7)
C15	0.0285 (9)	0.0292 (9)	0.0252 (9)	0.0038 (7)	0.0037 (7)	0.0009 (7)

Geometric parameters (Å, º)

O1—C1	1.250 (2)	C7—C8	1.517 (3)
O2—C3	1.231 (2)	C7—H7A	0.9900
N1—C3	1.393 (2)	C7—H7B	0.9900
N1—C10	1.408 (2)	C8—C9	1.513 (3)
N1—N2	1.4078 (19)	C8—H8A	0.9900
N2—C1	1.366 (2)	C8—H8B	0.9900
N2—H2	0.94 (2)	C9—H9A	0.9900
N3—C4	1.318 (2)	C9—H9B	0.9900
N3—C5	1.468 (2)	C10—C15	1.396 (2)
N3—C9	1.470 (2)	C10—C11	1.396 (2)
C1—C2	1.448 (2)	C11—C12	1.389 (2)
C2—C4	1.388 (2)	C11—H11	0.9500
C2—C3	1.454 (2)	C12—C13	1.387 (3)
C4—H4	0.9500	C12—H12	0.9500

C5—C6	1.510 (3)	C13—C14	1.383 (3)
C5—H5A	0.9900	C13—H13	0.9500
C5—H5B	0.9900	C14—C15	1.389 (2)
C6—C7	1.515 (3)	C14—H14	0.9500
C6—H6A	0.9900	C15—H15	0.9500
C6—H6B	0.9900		

C3—N1—C10	128.69 (14)	C8—C7—H7A	109.7
C3—N1—N2	109.25 (13)	C6—C7—H7B	109.7
C10—N1—N2	118.48 (13)	C8—C7—H7B	109.7
C1—N2—N1	109.92 (13)	H7A—C7—H7B	108.2
C1—N2—H2	124.2 (13)	C9—C8—C7	111.39 (16)
N1—N2—H2	115.4 (13)	C9—C8—H8A	109.4
C4—N3—C5	124.23 (15)	C7—C8—H8A	109.4
C4—N3—C9	120.63 (15)	C9—C8—H8B	109.4
C5—N3—C9	115.13 (14)	C7—C8—H8B	109.4
O1—C1—N2	121.49 (15)	H8A—C8—H8B	108.0
O1—C1—C2	131.76 (15)	N3—C9—C8	111.15 (16)
N2—C1—C2	106.70 (14)	N3—C9—H9A	109.4
C4—C2—C1	133.42 (16)	C8—C9—H9A	109.4
C4—C2—C3	118.37 (15)	N3—C9—H9B	109.4
C1—C2—C3	107.60 (14)	C8—C9—H9B	109.4
O2—C3—N1	124.67 (15)	H9A—C9—H9B	108.0
O2—C3—C2	129.46 (15)	C15—C10—C11	120.53 (16)
N1—C3—C2	105.81 (14)	C15—C10—N1	119.54 (15)
N3—C4—C2	130.85 (16)	C11—C10—N1	119.88 (15)
N3—C4—H4	114.6	C12—C11—C10	119.13 (17)
C2—C4—H4	114.6	C12—C11—H11	120.4
N3—C5—C6	110.34 (15)	C10—C11—H11	120.4
N3—C5—H5A	109.6	C13—C12—C11	120.89 (18)
C6—C5—H5A	109.6	C13—C12—H12	119.6
N3—C5—H5B	109.6	C11—C12—H12	119.6
C6—C5—H5B	109.6	C14—C13—C12	119.30 (17)
H5A—C5—H5B	108.1	C14—C13—H13	120.3
C5—C6—C7	111.58 (17)	C12—C13—H13	120.3
C5—C6—H6A	109.3	C13—C14—C15	121.20 (18)
C7—C6—H6A	109.3	C13—C14—H14	119.4
C5—C6—H6B	109.3	C15—C14—H14	119.4
C7—C6—H6B	109.3	C14—C15—C10	118.93 (17)
H6A—C6—H6B	108.0	C14—C15—H15	120.5
C6—C7—C8	109.83 (16)	C10—C15—H15	120.5
C6—C7—H7A	109.7		

C3—N1—N2—C1	−8.90 (19)	C4—N3—C5—C6	−125.26 (19)
C10—N1—N2—C1	−169.40 (14)	C9—N3—C5—C6	53.8 (2)
N1—N2—C1—O1	−169.98 (15)	N3—C5—C6—C7	−54.8 (2)
N1—N2—C1—C2	7.91 (18)	C5—C6—C7—C8	56.4 (2)
O1—C1—C2—C4	2.8 (3)	C6—C7—C8—C9	−55.3 (2)

N2—C1—C2—C4	−174.77 (18)	C4—N3—C9—C8	125.88 (18)
O1—C1—C2—C3	173.40 (17)	C5—N3—C9—C8	−53.2 (2)
N2—C1—C2—C3	−4.19 (18)	C7—C8—C9—N3	53.1 (2)
C10—N1—C3—O2	−13.6 (3)	C3—N1—C10—C15	42.5 (2)
N2—N1—C3—O2	−171.57 (16)	N2—N1—C10—C15	−161.32 (15)
C10—N1—C3—C2	163.79 (16)	C3—N1—C10—C11	−139.96 (18)
N2—N1—C3—C2	5.86 (18)	N2—N1—C10—C11	16.2 (2)
C4—C2—C3—O2	−11.6 (3)	C15—C10—C11—C12	−1.1 (3)
C1—C2—C3—O2	176.20 (18)	N1—C10—C11—C12	−178.65 (16)
C4—C2—C3—N1	171.17 (15)	C10—C11—C12—C13	0.1 (3)
C1—C2—C3—N1	−1.07 (18)	C11—C12—C13—C14	0.4 (3)
C5—N3—C4—C2	−11.1 (3)	C12—C13—C14—C15	0.0 (3)
C9—N3—C4—C2	169.92 (18)	C13—C14—C15—C10	−1.0 (3)
C1—C2—C4—N3	−21.8 (3)	C11—C10—C15—C14	1.5 (3)
C3—C2—C4—N3	168.43 (17)	N1—C10—C15—C14	179.07 (16)

Hydrogen-bond geometry (Å, º)

Cg3 is a centroid of the C10–C15 phenyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.94 (2)	1.86 (2)	2.8008 (18)	171 (2)
C5—H5*B*···O1	0.99	2.24	2.979 (2)	131
C9—H9*A*···O1^{ii}	0.99	2.51	3.421 (2)	152
C11—H11···O2^{iii}	0.95	2.52	3.471 (2)	178
C9—H9*B*···*Cg*3^{iv}	0.99	2.91	3.741 (2)	143

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