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2-Methyl-2-phenyl-1,2-dihydroquinazolin-4(3*H*)-one

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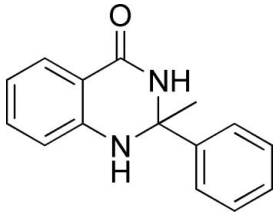
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.101; data-to-parameter ratio = 16.5.

In the molecule of the title compound, $C_{15}H_{14}N_2O$, the six-membered 1,3-diaza ring assumes an envelope conformation. The two benzene ring planes are almost perpendicular to each other, making a dihedral angle of 85.53 (5)°. Supramolecular aggregation is mainly effected by N—H···O hydrogen bonding.

Related literature

For general background, see: Jackson *et al.* (2007). For related structures, see: Shi *et al.* (2003, 2004); Yu *et al.* (1992).



Experimental

Crystal data

$C_{15}H_{14}N_2O$
M_r = 238.28
Monoclinic, $P2_1/n$
a = 8.4891 (7) Å
b = 8.7741 (8) Å
c = 16.1351 (16) Å
β = 93.543 (7)°
V = 1199.51 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 113 (2) K
0.28 × 0.24 × 0.22 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: none
14326 measured reflections
2835 independent reflections
2459 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.101
S = 1.11
2835 reflections
172 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 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
N1—H1···O1^{i}	0.878 (16)	1.970 (16)	2.8456 (12)	174.4 (13)

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

Data collection: *CrystalClear* (Rigaku, 2004); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

We thank Beijing Institute of Technology for financial support and Nankai University for the X-ray diffraction analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2381).

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supporting information

Acta Cryst. (2008). E**64**, o450 [doi:10.1107/S1600536807065427]

2-Methyl-2-phenyl-1,2-dihydroquinazolin-4(3*H*)-one

Lijun Zhang, Jiarong Li, Xiquan Yang, Daxin Shi and Jinnan Chen

S1. Comment

1,2-Dihydroquinazolin-4(3*H*)-ones are much important and useful nitrogen-containing heterocycles due to their diverse biological activities. They have been widely used as antitumors, α-adrenoceptors antagonists, diuretics, herbicides and plant growth relugators (Jackson *et al.*, 2007). The present investigation is aimed at the study of the molecular and supramolecular architecture of the title compound, (I), and may serve as a forerunner to a study of the correlation of these features with its biological activity.

The molecular structure of (I) is shown in Fig. 1. The bond distances and angles (Table 1) agree with those found in a reported 1,2-dihydroquinazolin-4(3*H*)-ones (Shi *et al.*, 2004). The 1,3-diaza ring exists in an envelope conformation, similar to that found in 4(3*H*)-quinazolinone derivatives (Yu *et al.*, 1992; Shi *et al.*, 2003). Two phenyl planes are almost perpendicular to each other with an angle of 85.53 (5)°. The O1 atom is deviated from C2-phenyl plane with 0.4706 Å. The crystal structure is stabilized by N—H···O interactions (Table 2, Fig. 2).

S2. Experimental

To a solution of DMF (10 ml) and $ZnCl_2$ (6 mmol) were added substituted 2-aminobenzonitrile (6 mmol) and acetophenone (6 mmol). The mixture was heated at reflux for 3 h. After completion of the reaction as indicated by TLC (eluent: ethyl acetate), the cooled reaction mixture was quenched with water and the precipitate was separated by filtration. The filtration residue was dispersed into water and titrated to pH 12–13 by 20% sodium hydroxide. After filtration, the product was isolated by column chromatography (200–300 mesh silica gel, ethyl acetate-petroleum with 1:2); yield 62%. Single crystals were obtained from an ethanol solution by slow evaporation at room temperature.

S3. Refinement

Imino H atoms were located in a difference Fourier map and refined isotropically. Other H atoms were placed inca lucalculated positions with C—H = 0.95 (aromatic) or 0.98 Å (methyl), and refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) (for methyl group).

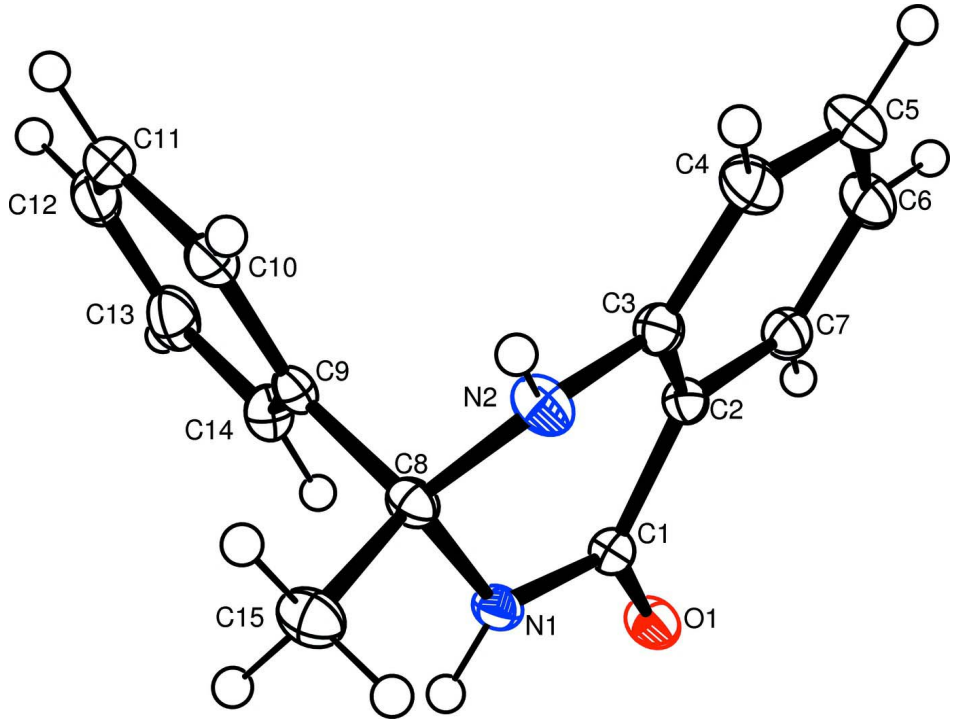

Figure 1

The molecular structure of (I), with 30% probability displacement ellipsoids for non-H atoms.

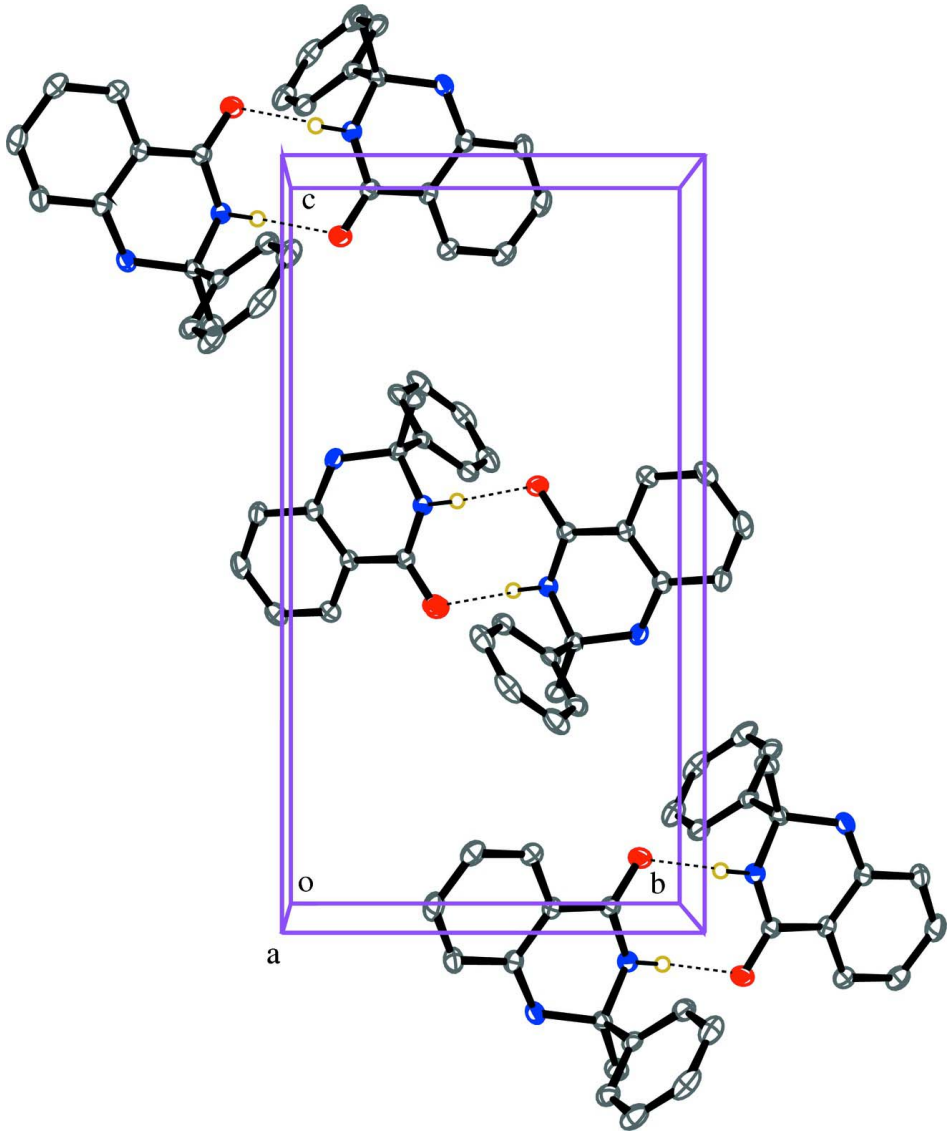

Figure 2

The packing of (I), viewed down the *a* axis, showing one layer of molecules connected by N—H···O hydrogen bonds (dashed lines).

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Crystal data

$C_{15}H_{14}N_2O$
$M_r = 238.28$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.4891\ (7)$ Å
$b = 8.7741\ (8)$ Å
$c = 16.1351\ (16)$ Å
$\beta = 93.543\ (7)°$
$V = 1199.51\ (19)$ Å^3
$Z = 4$

$F(000) = 504$
$D_x = 1.319$ Mg m^{-3}
Melting point = 505–507 K
Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
Cell parameters from 3772 reflections
$\theta = 2.6–27.9°$
$\mu = 0.08$ mm^{-1}
$T = 113$ K
Prism, colorless
0.28 × 0.24 × 0.22 mm

Data collection

Rigaku Saturn diffractometer
Radiation source: rotating anode
Confocal monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω scans
14326 measured reflections
2835 independent reflections
2459 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.6°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -20 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.101$
$S = 1.11$
2835 reflections
172 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
$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 0.2057P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Experimental. IR (KBr, cm^{-1}): 3389, 3181, 1663, 1613; 1H NMR (DMSO-d$_6$, 400 MHz) δ_H: 1.79 (3*H*, s, CH$_3$), 6.63–6.67 (1*H*, m, J=0.8, 8.0 Hz, ArH), 6.83 (1*H*, t, J=0.8, 8.0 Hz, ArH), 6.89 (1*H*, s, NH), 7.21–7.23 (2*H*, m, ArH), 7.28–7.32 (2*H*, m, ArH), 7.61–7.68 (3*H*, m, ArH), 7.93 (1*H*, s, NH); 13 C NMR (DMSO-d$_6$, 100 MHz) δ_C: 31.14, 71.54, 115.36, 116.67, 118.26, 126.09 (2 C), 128.00, 128.45, 128.87 (2 C), 134.05, 147.95, 148.23, 164.90; MS (ESI): m/*z* (%) =239.1 (100) $[M+H]^+$; $C_{15}H_{14}N_2O$: calcd. C 75.61, H 5.92, N 11.76; found C 75.28, H 6.11, N 11.43.

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	0.41106 (9)	0.13177 (9)	−0.08264 (4)	0.0236 (2)
N1	0.44850 (10)	0.16028 (10)	0.05706 (5)	0.0173 (2)
N2	0.38757 (11)	0.38938 (10)	0.12264 (6)	0.0208 (2)
C1	0.38879 (12)	0.20563 (11)	−0.01835 (6)	0.0168 (2)
C2	0.30141 (12)	0.35144 (11)	−0.02015 (6)	0.0165 (2)
C3	0.30957 (12)	0.44441 (11)	0.05098 (6)	0.0180 (2)
C4	0.24352 (13)	0.59131 (12)	0.04600 (7)	0.0245 (2)
H4	0.2517	0.6571	0.0928	0.029*
C5	0.16659 (14)	0.63954 (13)	−0.02730 (8)	0.0285 (3)
H5	0.1215	0.7387	−0.0302	0.034*
C6	0.15378 (13)	0.54548 (13)	−0.09718 (7)	0.0261 (3)
H6	0.0983	0.5792	−0.1467	0.031*

C7	0.22271 (12)	0.40276 (12)	−0.09347 (6)	0.0206 (2)
H7	0.2165	0.3390	−0.1412	0.025*
C8	0.39390 (12)	0.22450 (11)	0.13376 (6)	0.0170 (2)
C9	0.23069 (12)	0.15939 (11)	0.15239 (6)	0.0174 (2)
C10	0.15071 (13)	0.22173 (13)	0.21771 (7)	0.0236 (2)
H10	0.1973	0.3033	0.2493	0.028*
C11	0.00388 (15)	0.16596 (14)	0.23708 (8)	0.0304 (3)
H11	−0.0489	0.2090	0.2818	0.036*
C12	−0.06521 (14)	0.04741 (14)	0.19100 (8)	0.0321 (3)
H12	−0.1659	0.0099	0.2036	0.038*
C13	0.01311 (14)	−0.01636 (14)	0.12637 (8)	0.0289 (3)
H13	−0.0340	−0.0977	0.0949	0.035*
C14	0.16110 (13)	0.03889 (12)	0.10750 (7)	0.0220 (2)
H14	0.2147	−0.0061	0.0637	0.026*
C15	0.51453 (13)	0.18452 (13)	0.20463 (7)	0.0234 (2)
H15A	0.6170	0.2290	0.1935	0.035*
H15B	0.5247	0.0735	0.2089	0.035*
H15C	0.4792	0.2254	0.2569	0.035*
H1	0.4966 (17)	0.0719 (18)	0.0626 (9)	0.036 (4)*
H2	0.3890 (18)	0.4458 (18)	0.1676 (9)	0.040 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0313 (4)	0.0223 (4)	0.0173 (4)	0.0078 (3)	0.0024 (3)	−0.0011 (3)
N1	0.0186 (4)	0.0162 (4)	0.0171 (4)	0.0044 (3)	0.0014 (3)	0.0000 (3)
N2	0.0263 (5)	0.0155 (4)	0.0201 (5)	0.0012 (4)	−0.0037 (4)	−0.0032 (3)
C1	0.0162 (5)	0.0168 (5)	0.0177 (5)	0.0000 (4)	0.0017 (4)	0.0010 (4)
C2	0.0153 (5)	0.0149 (5)	0.0194 (5)	0.0000 (4)	0.0017 (4)	0.0018 (4)
C3	0.0164 (5)	0.0161 (5)	0.0214 (5)	−0.0014 (4)	0.0017 (4)	0.0013 (4)
C4	0.0292 (6)	0.0161 (5)	0.0285 (6)	0.0017 (4)	0.0026 (5)	−0.0017 (4)
C5	0.0316 (6)	0.0180 (5)	0.0361 (7)	0.0071 (4)	0.0030 (5)	0.0049 (5)
C6	0.0262 (6)	0.0255 (6)	0.0264 (6)	0.0049 (4)	−0.0018 (5)	0.0079 (4)
C7	0.0197 (5)	0.0214 (5)	0.0207 (5)	0.0001 (4)	0.0007 (4)	0.0021 (4)
C8	0.0197 (5)	0.0154 (5)	0.0154 (5)	0.0027 (4)	−0.0010 (4)	−0.0011 (4)
C9	0.0189 (5)	0.0176 (5)	0.0155 (5)	0.0045 (4)	−0.0007 (4)	0.0047 (4)
C10	0.0275 (6)	0.0226 (5)	0.0208 (5)	0.0083 (4)	0.0032 (4)	0.0048 (4)
C11	0.0305 (6)	0.0319 (6)	0.0302 (6)	0.0140 (5)	0.0128 (5)	0.0137 (5)
C12	0.0212 (6)	0.0344 (7)	0.0412 (7)	0.0046 (5)	0.0065 (5)	0.0215 (6)
C13	0.0259 (6)	0.0267 (6)	0.0336 (6)	−0.0049 (5)	−0.0024 (5)	0.0098 (5)
C14	0.0239 (5)	0.0220 (5)	0.0200 (5)	−0.0008 (4)	0.0007 (4)	0.0030 (4)
C15	0.0241 (5)	0.0246 (6)	0.0206 (5)	0.0044 (4)	−0.0051 (4)	−0.0021 (4)

Geometric parameters (Å, º)

O1—C1	1.2473 (12)	C7—H7	0.9500
N1—C1	1.3489 (13)	C8—C15	1.5279 (14)
N1—C8	1.4612 (13)	C8—C9	1.5452 (14)

N1—H1	0.878 (16)	C9—C14	1.3924 (15)
N2—C3	1.3834 (13)	C9—C10	1.3997 (15)
N2—C8	1.4583 (13)	C10—C11	1.3925 (17)
N2—H2	0.878 (15)	C10—H10	0.9500
C1—C2	1.4782 (13)	C11—C12	1.3874 (19)
C2—C7	1.3968 (14)	C11—H11	0.9500
C2—C3	1.4062 (14)	C12—C13	1.3886 (18)
C3—C4	1.4059 (15)	C12—H12	0.9500
C4—C5	1.3816 (16)	C13—C14	1.3976 (16)
C4—H4	0.9500	C13—H13	0.9500
C5—C6	1.3961 (17)	C14—H14	0.9500
C5—H5	0.9500	C15—H15A	0.9800
C6—C7	1.3820 (15)	C15—H15B	0.9800
C6—H6	0.9500	C15—H15C	0.9800

C1—N1—C8	121.94 (8)	N1—C8—C15	108.20 (8)
C1—N1—H1	119.9 (9)	N2—C8—C9	111.35 (8)
C8—N1—H1	115.1 (9)	N1—C8—C9	110.94 (8)
C3—N2—C8	117.47 (9)	C15—C8—C9	109.70 (8)
C3—N2—H2	118.4 (10)	C14—C9—C10	118.59 (10)
C8—N2—H2	117.3 (10)	C14—C9—C8	122.43 (9)
O1—C1—N1	121.94 (9)	C10—C9—C8	118.98 (9)
O1—C1—C2	122.35 (9)	C11—C10—C9	120.95 (11)
N1—C1—C2	115.66 (9)	C11—C10—H10	119.5
C7—C2—C3	120.07 (9)	C9—C10—H10	119.5
C7—C2—C1	120.58 (9)	C12—C11—C10	119.85 (11)
C3—C2—C1	119.13 (9)	C12—C11—H11	120.1
N2—C3—C4	122.43 (10)	C10—C11—H11	120.1
N2—C3—C2	118.46 (9)	C11—C12—C13	119.90 (11)
C4—C3—C2	119.07 (10)	C11—C12—H12	120.1
C5—C4—C3	119.70 (10)	C13—C12—H12	120.1
C5—C4—H4	120.2	C12—C13—C14	120.16 (11)
C3—C4—H4	120.2	C12—C13—H13	119.9
C4—C5—C6	121.29 (10)	C14—C13—H13	119.9
C4—C5—H5	119.4	C9—C14—C13	120.55 (10)
C6—C5—H5	119.4	C9—C14—H14	119.7
C7—C6—C5	119.27 (10)	C13—C14—H14	119.7
C7—C6—H6	120.4	C8—C15—H15A	109.5
C5—C6—H6	120.4	C8—C15—H15B	109.5
C6—C7—C2	120.53 (10)	H15A—C15—H15B	109.5
C6—C7—H7	119.7	C8—C15—H15C	109.5
C2—C7—H7	119.7	H15A—C15—H15C	109.5
N2—C8—N1	106.82 (8)	H15B—C15—H15C	109.5
N2—C8—C15	109.74 (8)		

C8—N1—C1—O1	−165.49 (9)	C3—N2—C8—C15	167.04 (9)
C8—N1—C1—C2	17.02 (14)	C3—N2—C8—C9	−71.30 (11)
O1—C1—C2—C7	8.20 (15)	C1—N1—C8—N2	−45.85 (12)

N1—C1—C2—C7	−174.31 (9)	C1—N1—C8—C15	−163.94 (9)
O1—C1—C2—C3	−166.39 (10)	C1—N1—C8—C9	75.67 (12)
N1—C1—C2—C3	11.10 (14)	N2—C8—C9—C14	127.89 (10)
C8—N2—C3—C4	155.35 (10)	N1—C8—C9—C14	9.06 (13)
C8—N2—C3—C2	−26.98 (14)	C15—C8—C9—C14	−110.43 (11)
C7—C2—C3—N2	179.56 (9)	N2—C8—C9—C10	−53.28 (12)
C1—C2—C3—N2	−5.82 (14)	N1—C8—C9—C10	−172.12 (9)
C7—C2—C3—C4	−2.69 (15)	C15—C8—C9—C10	68.40 (11)
C1—C2—C3—C4	171.93 (9)	C14—C9—C10—C11	−0.68 (15)
N2—C3—C4—C5	−179.80 (10)	C8—C9—C10—C11	−179.55 (9)
C2—C3—C4—C5	2.54 (16)	C9—C10—C11—C12	−0.31 (16)
C3—C4—C5—C6	−0.44 (18)	C10—C11—C12—C13	0.76 (17)
C4—C5—C6—C7	−1.56 (18)	C11—C12—C13—C14	−0.19 (17)
C5—C6—C7—C2	1.42 (17)	C10—C9—C14—C13	1.25 (15)
C3—C2—C7—C6	0.71 (16)	C8—C9—C14—C13	−179.92 (9)
C1—C2—C7—C6	−173.83 (10)	C12—C13—C14—C9	−0.82 (17)
C3—N2—C8—N1	49.96 (11)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.878 (16)	1.970 (16)	2.8456 (12)	174.4 (13)

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