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6-Methoxy-2,3,4,9-tetrahydro-1*H*-carbazol-1-one

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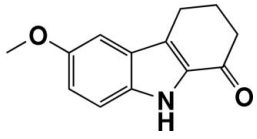
Key indicators: single-crystal X-ray study; T = 160 K; mean σ(C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.146; data-to-parameter ratio = 20.7.

The carbazole unit of the title molecule, $C_{13}H_{13}NO_2$, is not planar. The dihedral angle between the benzene ring and the pyrrole ring is 1.69 (6)°. The cyclohexene ring adopts an envelope conformation. Intermolecular C—H···O and N—H···O hydrogen bonds are present in the crystal structure. A C—H···π interaction, involving the benzene ring, is also found in the crystal structure.

Related literature

For related literature, see: Bhattacharya & Chakraborty (1987); Chakraborty & Roy (1991); Chakraborty (1993); Knolker (1986); Lescot *et al.* (1986); Hook *et al.* (1990); Hirata *et al.* (1999); Kapil (1971); Knolker & Reddy (2002); Sowmithran & Rajendra Prasad (1986); Rajendra Prasad & Vijayalakshmi (1994). Gunaseelan *et al.* (2007*a*,*b*) and Thiruvalluvar *et al.* (2007) have reported the crystal structures of substituted carbazole derivatives, in which the carbazole units are not planar.



Experimental

Crystal data

$C_{13}H_{13}NO_2$
M_r = 215.24
Monoclinic, $P2_1/c$
a = 9.0627 (2) Å
b = 14.0285 (3) Å
c = 8.5506 (2) Å
β = 101.815 (1)°
V = 1064.06 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 160 (1) K
0.35 × 0.28 × 0.13 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
28554 measured reflections
3077 independent reflections
2601 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.145
S = 1.12
3077 reflections
149 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 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
N9—H9···O1^{i}	0.948 (17)	1.918 (17)	2.8313 (14)	161.2 (15)
C2—H2A···O2^{ii}	0.99	2.52	3.4962 (15)	169
C4—H4B···Cg^{iii}	0.99	2.57	3.492 (1)	156

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+1, y, z+1$; (iii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$. Cg is the centroid of the benzene ring.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); 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: WN2245).

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6-Methoxy-2,3,4,9-tetrahydro-1*H*-carbazol-1-one

M. Sridharan, K. J. Rajendra Prasad, A. Thomas Gunaseelan, A. Thiruvalluvar and A. Linden

S1. Comment

Heterocylic compounds are encountered in a very large number of groups of organic compounds. They play a vital role in the metabolism of all living cells, which are widely distributed in nature and are essential to life. Among them the carbazole heterocycles have emerged as an important class, based on their fascinating structure and high degree of biological activities (Bhattacharya & Chakraborty,1987; Chakraborty & Roy, 1991; Chakraborty, 1993). A number of carbazole alkaloids with intriguing novel structures and useful biological activities were isolated from natural sources over the past decades; these attracted chemists to frame novel synthetic strategies towards the synthesis of carbazole and its derivatives (Knolker,1986; Lescot *et al.*, 1986). These alkaloids represent a new and interesting variant in the large number of indole alkaloids, which have yielded several important drugs. Several reports have appeared on the synthesis of carbazole derivatives, in connection with the search for newer physiologically active compounds (Hook *et al.*, 1990; Hirata *et al.*, 1999; Kapil, 1971; Knolker & Reddy, 2002). The preparation of 1-oxo compounds via their corresponding hydrazones have been reported (Sowmithran & Rajendra Prasad, 1986; Rajendra Prasad & Vijayalakshmi, 1994).

Gunaseelan *et al.* (2007*a*,b) and Thiruvalluvar *et al.* (2007) have reported the crystal structures of substituted carbazole derivatives, in which the carbazole units are not planar. The molecular structure of the title compound, with atomic numbering scheme, is shown in Fig. 1. The carbazole unit of the title molecule is not planar. The dihedral angle between the benzene ring and the pyrrole ring is 1.69 (6)°. The cyclohexene ring adopts an envelope conformation. Intermolecular C2—H2A···O2(x + 1, y, z + 1) and N9—H9···O1(-x + 1, -y + 1, -z + 1) hydrogen bonds are present in the crystal structure (Fig. 2). A C4—H4B···π(x, 3/2 - y,1/2 + z) interaction involving the benzene ring is also found in the structure, .

S2. Experimental

A solution of 2-(2-(4-methoxyphenyl)hydrazono)cyclohexanone (232 mg, 0.001 mol) in a mixture of acetic acid (20 ml) and hydrochloric acid (5 ml) was refluxed on an oil bath pre-heated to 398-403 K for 2 h. The reaction was monitored by TLC. After completion of the reaction the contents were cooled and poured on to cold water with stirring. The brown solid which separated was purified by passing through a column of silica gel and eluting with a (95:5) petroleum ether-ethyl acetate mixture, yielding the title compound (144 mg, 67%). The compound thus obtained was recrystallized using ethanol.

S3. Refinement

The H atom bonded to N9 was located in a difference Fourier map and refined isotropically. Other H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.95–0.99 Å and U_{iso}(H) = xU_{eq}(parent atom), where x = 1.5 for methyl and 1.2 for all other carbon-bound H atoms.

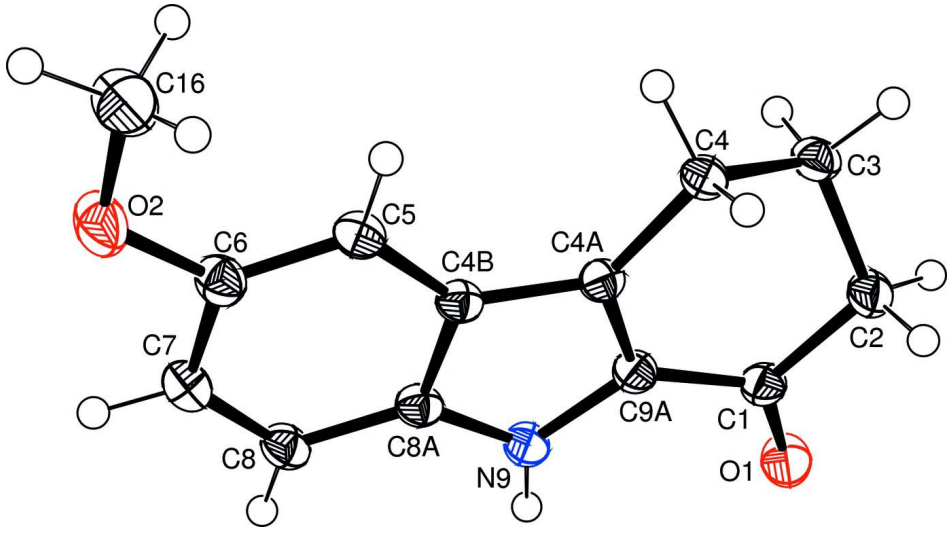

Figure 1

The molecular structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level. Hydrogen atoms are represented by spheres of arbitrary radius.

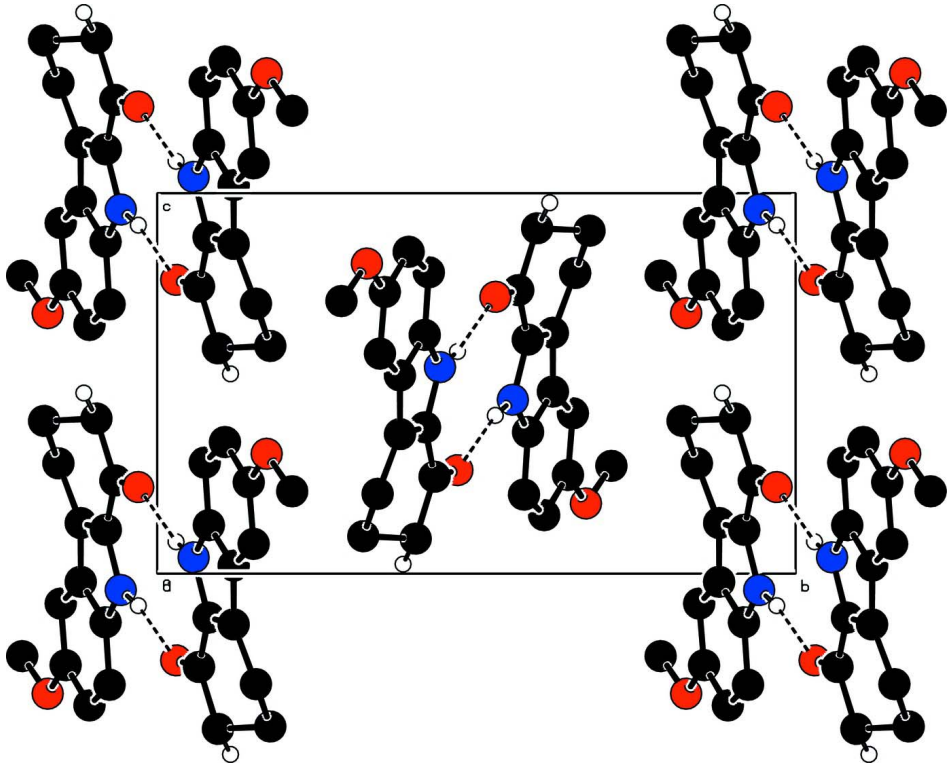

Figure 2

The molecular packing of the title compound, viewed down the *a* axis. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

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Crystal data

$C_{13}H_{13}NO_2$
$M_r = 215.24$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.0627$ (2) Å
$b = 14.0285$ (3) Å
$c = 8.5506$ (2) Å
$\beta = 101.815$ (1)°
$V = 1064.06$ (4) Å^3
$Z = 4$
$F(000) = 456$
$D_x = 1.344$ Mg m^{-3}
Melting point: 536 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3175 reflections
$\theta = 2.0$–$30.0°$
$\mu = 0.09$ mm^{-1}
$T = 160$ K
Tablet, colourless
0.35 × 0.28 × 0.13 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Radiation source: Nonius FR590 sealed tube generator
Horizontally mounted graphite crystal monochromator
Detector resolution: 9 pixels mm^{-1}
φ and ω scans with κ offsets
28554 measured reflections
3077 independent reflections
2601 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 30.0°$, $\theta_{min} = 2.3°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.146$
$S = 1.12$
3077 reflections
149 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
$w = 1/[\sigma^2(F_o^2) + (0.0825P)^2 + 0.2332P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Special details

Experimental. Solvent used: EtOH Cooling Device: Oxford Cryosystems Cryostream 700 Crystal mount: glued on a glass fibre Mosaicity (°.): 0.742 (2) Frames collected: 359 Seconds exposure per frame: 100 Degrees rotation per frame: 2.0 Crystal-Detector distance (mm): 30.0

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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	0.53223 (10)	0.52978 (7)	0.72771 (11)	0.0324 (3)
O2	−0.29638 (10)	0.67151 (8)	0.18475 (11)	0.0354 (3)

N9	0.27900 (11)	0.55709 (7)	0.45710 (11)	0.0229 (3)
C1	0.41200 (13)	0.56555 (8)	0.74513 (13)	0.0229 (3)
C2	0.38660 (13)	0.59515 (9)	0.90781 (13)	0.0251 (3)
C3	0.27324 (13)	0.67715 (8)	0.90149 (13)	0.0236 (3)
C4	0.12214 (12)	0.65469 (8)	0.79020 (12)	0.0213 (3)
C4A	0.14868 (12)	0.61976 (7)	0.63321 (12)	0.0196 (3)
C4B	0.05307 (12)	0.61980 (7)	0.47874 (13)	0.0197 (3)
C5	−0.09638 (12)	0.65107 (8)	0.42156 (13)	0.0219 (3)
C6	−0.15394 (12)	0.64344 (8)	0.25953 (13)	0.0241 (3)
C7	−0.06690 (13)	0.60611 (8)	0.15394 (13)	0.0253 (3)
C8	0.07867 (13)	0.57456 (8)	0.20802 (13)	0.0236 (3)
C8A	0.13856 (12)	0.58090 (7)	0.37241 (13)	0.0208 (3)
C9A	0.28448 (12)	0.58046 (8)	0.61527 (13)	0.0213 (3)
C16	−0.38951 (15)	0.71146 (13)	0.28342 (18)	0.0441 (5)
H2A	0.48420	0.61484	0.97491	0.0301*
H2B	0.34978	0.53939	0.95963	0.0301*
H3A	0.25628	0.68946	1.01044	0.0283*
H3B	0.31607	0.73572	0.86390	0.0283*
H4A	0.06803	0.60539	0.83938	0.0256*
H4B	0.05882	0.71278	0.77373	0.0256*
H5	−0.15509	0.67641	0.49192	0.0262*
H7	−0.10988	0.60272	0.04300	0.0303*
H8	0.13633	0.54942	0.13648	0.0284*
H9	0.3587 (19)	0.5296 (12)	0.416 (2)	0.038 (4)*
H16A	−0.48742	0.72886	0.21742	0.0661*
H16B	−0.34089	0.76845	0.33697	0.0661*
H16C	−0.40428	0.66454	0.36374	0.0661*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0269 (5)	0.0436 (5)	0.0274 (4)	0.0137 (4)	0.0075 (3)	−0.0016 (4)
O2	0.0230 (4)	0.0513 (6)	0.0293 (5)	0.0061 (4)	−0.0008 (3)	−0.0103 (4)
N9	0.0239 (5)	0.0262 (5)	0.0202 (4)	0.0049 (3)	0.0084 (3)	−0.0009 (3)
C1	0.0245 (5)	0.0232 (5)	0.0222 (5)	0.0045 (4)	0.0076 (4)	0.0004 (4)
C2	0.0249 (5)	0.0302 (6)	0.0205 (5)	0.0067 (4)	0.0054 (4)	−0.0010 (4)
C3	0.0235 (5)	0.0252 (5)	0.0227 (5)	0.0028 (4)	0.0064 (4)	−0.0045 (4)
C4	0.0224 (5)	0.0233 (5)	0.0196 (5)	0.0030 (4)	0.0073 (4)	−0.0010 (4)
C4A	0.0215 (5)	0.0188 (5)	0.0199 (5)	0.0012 (3)	0.0073 (4)	0.0012 (3)
C4B	0.0215 (5)	0.0182 (5)	0.0205 (5)	−0.0004 (4)	0.0071 (4)	−0.0004 (3)
C5	0.0212 (5)	0.0223 (5)	0.0232 (5)	−0.0014 (4)	0.0071 (4)	−0.0022 (4)
C6	0.0208 (5)	0.0260 (5)	0.0249 (5)	−0.0018 (4)	0.0036 (4)	−0.0033 (4)
C7	0.0274 (6)	0.0272 (5)	0.0211 (5)	−0.0025 (4)	0.0045 (4)	−0.0034 (4)
C8	0.0276 (5)	0.0245 (5)	0.0204 (5)	−0.0009 (4)	0.0089 (4)	−0.0025 (4)
C8A	0.0231 (5)	0.0200 (5)	0.0210 (5)	0.0001 (4)	0.0085 (4)	−0.0005 (3)
C9A	0.0232 (5)	0.0222 (5)	0.0197 (5)	0.0030 (4)	0.0073 (4)	0.0002 (4)
C16	0.0261 (6)	0.0610 (10)	0.0421 (8)	0.0118 (6)	0.0000 (5)	−0.0185 (7)

Geometric parameters (Å, º)

O1—C1	1.2360 (15)	C6—C7	1.4154 (16)
O2—C6	1.3756 (15)	C7—C8	1.3785 (17)
O2—C16	1.4247 (18)	C8—C8A	1.4021 (15)
N9—C8A	1.3706 (15)	C2—H2A	0.9900
N9—C9A	1.3826 (14)	C2—H2B	0.9900
N9—H9	0.948 (17)	C3—H3A	0.9900
C1—C9A	1.4446 (16)	C3—H3B	0.9900
C1—C2	1.5138 (16)	C4—H4A	0.9900
C2—C3	1.5359 (17)	C4—H4B	0.9900
C3—C4	1.5318 (16)	C5—H5	0.9500
C4—C4A	1.4940 (14)	C7—H7	0.9500
C4A—C4B	1.4236 (15)	C8—H8	0.9500
C4A—C9A	1.3854 (16)	C16—H16A	0.9800
C4B—C8A	1.4189 (15)	C16—H16B	0.9800
C4B—C5	1.4124 (16)	C16—H16C	0.9800
C5—C6	1.3810 (15)		
O1···N9	2.9314 (13)	C16···H3B^{viii}	2.9800
O1···N9^{i}	2.8313 (14)	H2A···O2^{x}	2.5200
O1···H9	2.804 (17)	H2A···C16^{x}	2.9800
O1···H2B^{ii}	2.8400	H2A···H16A^{x}	2.5900
O1···H9^{i}	1.918 (17)	H2B···O1^{ii}	2.8400
O2···H2A^{iii}	2.5200	H2B···C2^{ii}	3.0700
N9···O1	2.9314 (13)	H3A···C8^{xi}	3.0300
N9···O1^{i}	2.8313 (14)	H3A···H8^{xi}	2.5900
C1···C16^{iv}	3.590 (2)	H3B···C9A	3.0200
C2···C2^{ii}	3.5370 (17)	H3B···C8A^{v}	3.0400
C3···C8A^{v}	3.5983 (15)	H3B···C16^{iv}	2.9800
C4B···C4B^{vi}	3.5353 (14)	H3B···H16A^{iv}	2.4300
C6···C9A^{vi}	3.5958 (16)	H4A···C7^{vi}	2.9700
C8A···C3^{vii}	3.5983 (15)	H4A···C8^{vi}	2.8400
C9A···C6^{vi}	3.5958 (16)	H4B···C4B^{v}	2.9400
C16···C1^{viii}	3.590 (2)	H4B···C5^{v}	2.8200
C1···H16A^{iv}	3.0500	H4B···C6^{v}	2.7800
C1···H9^{i}	3.028 (17)	H4B···C7^{v}	2.8900
C2···H2B^{ii}	3.0700	H4B···C8^{v}	3.0500
C4B···H4B^{vii}	2.9400	H4B···C8A^{v}	3.0600
C5···H16C	2.7400	H5···C16	2.5300
C5···H4B^{vii}	2.8200	H5···H16B	2.3100
C5···H16B	2.7400	H5···H16C	2.3100
C6···H4B^{vii}	2.7800	H8···H3A^{ix}	2.5900
C7···H4B^{vii}	2.8900	H9···O1	2.804 (17)
C7···H4A^{vi}	2.9700	H9···O1^{i}	1.918 (17)
C8···H4B^{vii}	3.0500	H9···C1^{i}	3.028 (17)
C8···H4A^{vi}	2.8400	H16A···H2A^{iii}	2.5900
C8···H3A^{ix}	3.0300	H16A···C1^{viii}	3.0500

C8A···H4B^{vii}	3.0600	H16A···H3B^{viii}	2.4300
C8A···H3B^{vii}	3.0400	H16B···C5	2.7400
C9A···H3B	3.0200	H16B···H5	2.3100
C16···H2A^{iii}	2.9800	H16C···C5	2.7400
C16···H5	2.5300	H16C···H5	2.3100

C6—O2—C16	116.79 (10)	C1—C2—H2A	109.00
C8A—N9—C9A	107.61 (9)	C1—C2—H2B	109.00
C9A—N9—H9	125.6 (10)	C3—C2—H2A	109.00
C8A—N9—H9	126.8 (10)	C3—C2—H2B	109.00
O1—C1—C9A	123.53 (10)	H2A—C2—H2B	108.00
O1—C1—C2	121.72 (10)	C2—C3—H3A	109.00
C2—C1—C9A	114.73 (10)	C2—C3—H3B	109.00
C1—C2—C3	113.55 (9)	C4—C3—H3A	109.00
C2—C3—C4	111.98 (9)	C4—C3—H3B	109.00
C3—C4—C4A	109.74 (9)	H3A—C3—H3B	108.00
C4B—C4A—C9A	106.45 (9)	C3—C4—H4A	110.00
C4—C4A—C4B	130.85 (10)	C3—C4—H4B	110.00
C4—C4A—C9A	122.69 (10)	C4A—C4—H4A	110.00
C5—C4B—C8A	120.56 (10)	C4A—C4—H4B	110.00
C4A—C4B—C8A	106.61 (9)	H4A—C4—H4B	108.00
C4A—C4B—C5	132.82 (10)	C4B—C5—H5	121.00
C4B—C5—C6	117.50 (10)	C6—C5—H5	121.00
O2—C6—C5	124.74 (10)	C6—C7—H7	119.00
O2—C6—C7	113.70 (10)	C8—C7—H7	119.00
C5—C6—C7	121.55 (10)	C7—C8—H8	121.00
C6—C7—C8	121.66 (10)	C8A—C8—H8	121.00
C7—C8—C8A	117.60 (10)	O2—C16—H16A	109.00
N9—C8A—C8	129.88 (10)	O2—C16—H16B	109.00
C4B—C8A—C8	121.11 (10)	O2—C16—H16C	109.00
N9—C8A—C4B	108.97 (9)	H16A—C16—H16B	109.00
C1—C9A—C4A	124.16 (10)	H16A—C16—H16C	109.00
N9—C9A—C1	125.48 (10)	H16B—C16—H16C	109.00
N9—C9A—C4A	110.36 (10)		

C16—O2—C6—C5	0.21 (18)	C4B—C4A—C9A—N9	0.74 (12)
C16—O2—C6—C7	−178.89 (12)	C9A—C4A—C4B—C8A	−0.84 (11)
C9A—N9—C8A—C8	177.52 (11)	C4—C4A—C9A—N9	−178.17 (10)
C8A—N9—C9A—C4A	−0.35 (12)	C4—C4A—C4B—C8A	177.95 (10)
C9A—N9—C8A—C4B	−0.20 (12)	C9A—C4A—C4B—C5	−179.48 (11)
C8A—N9—C9A—C1	179.00 (10)	C5—C4B—C8A—N9	179.49 (10)
C9A—C1—C2—C3	−29.21 (14)	C4A—C4B—C8A—N9	0.65 (12)
O1—C1—C2—C3	152.17 (11)	C5—C4B—C8A—C8	1.54 (16)
C2—C1—C9A—N9	−178.39 (11)	C4A—C4B—C5—C6	177.60 (11)
C2—C1—C9A—C4A	0.87 (16)	C8A—C4B—C5—C6	−0.89 (16)
O1—C1—C9A—C4A	179.47 (11)	C4A—C4B—C8A—C8	−177.31 (10)
O1—C1—C9A—N9	0.21 (19)	C4B—C5—C6—O2	−179.27 (11)
C1—C2—C3—C4	54.63 (13)	C4B—C5—C6—C7	−0.24 (16)

C2—C3—C4—C4A	−49.23 (12)	O2—C6—C7—C8	179.92 (11)
C3—C4—C4A—C4B	−156.37 (11)	C5—C6—C7—C8	0.80 (18)
C3—C4—C4A—C9A	22.25 (14)	C6—C7—C8—C8A	−0.17 (17)
C4—C4A—C4B—C5	−0.7 (2)	C7—C8—C8A—N9	−178.45 (11)
C4B—C4A—C9A—C1	−178.62 (10)	C7—C8—C8A—C4B	−0.97 (16)
C4—C4A—C9A—C1	2.47 (17)		

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*+1, −*z*+2; (iii) *x*−1, *y*, *z*−1; (iv) *x*+1, −*y*+3/2, *z*+1/2; (v) *x*, −*y*+3/2, *z*+1/2; (vi) −*x*, −*y*+1, −*z*+1; (vii) *x*, −*y*+3/2, *z*−1/2; (viii) *x*−1, −*y*+3/2, *z*−1/2; (ix) *x*, *y*, *z*−1; (x) *x*+1, *y*, *z*+1; (xi) *x*, *y*, *z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N9—H9···O1^{i}	0.948 (17)	1.918 (17)	2.8313 (14)	161.2 (15)
C2—H2*A*···O2^{x}	0.99	2.52	3.4962 (15)	169
C4—H4*B*···*Cg*^{v}	0.99	2.57	3.492 (1)	156

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