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## Structure Reports

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## (E)-2-(Furan-2-ylmethylidene)-8-methyl-2,3,4,9-tetrahydro-1H-carbazol-1-one

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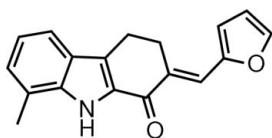
Received 3 November 2010; accepted 6 November 2010

 Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.050;  $wR$  factor = 0.157; data-to-parameter ratio = 14.0.

In the title molecule,  $\text{C}_{18}\text{H}_{15}\text{NO}_2$ , the carbazole unit is not planar [maximum deviation from mean plane = 0.236 (2) Å]. The pyrrole ring makes dihedral angles of 1.21 (10) and 16.74 (12)° with the benzene and the furan rings, respectively. The cyclohexene ring adopts a half-chair conformation. In the crystal, inversion dimers linked by pairs of  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds generate  $R_2^2(10)$  loops.

## Related literature

For the synthesis of hetero-annulated carbazoles, see: Knölker & Reddy (2002). For the derivation of various hetero-annulated carbazoles, see: Sridharan *et al.* (2008); Danish & Rajendra Prasad (2004, 2005); Periyasami *et al.* (2008, 2009). For ring conformations, see: Cremer & Pople (1975). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



## Experimental

## Crystal data

 $\text{C}_{18}\text{H}_{15}\text{NO}_2$   
 $M_r = 277.31$   
 Orthorhombic, *Pbca*  
 $a = 6.7353$  (1) Å

 $b = 16.1393$  (3) Å  
 $c = 25.9549$  (4) Å  
 $V = 2821.38$  (8) Å<sup>3</sup>  
 $Z = 8$ 

 Cu  $K\alpha$  radiation  
 $\mu = 0.68$  mm<sup>-1</sup>
 $T = 295$  K  
 $0.44 \times 0.28 \times 0.12$  mm

## Data collection

 Oxford Diffraction Xcalibur Ruby  
 Gemini diffractometer  
 Absorption correction: multi-scan  
 (*CrysAlis PRO*; Oxford  
 Diffraction, 2010)  
 $T_{\min} = 0.888$ ,  $T_{\max} = 1.000$ 

 6361 measured reflections  
 2736 independent reflections  
 2201 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.026$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.050$   
 $wR(F^2) = 0.157$   
 $S = 1.05$   
 2736 reflections  
 195 parameters

 H atoms treated by a mixture of  
 independent and constrained  
 refinement  
 $\Delta\rho_{\text{max}} = 0.23$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.26$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N9}-\text{H9}\cdots\text{O1}^i$	0.89 (3)	2.01 (3)	2.8969 (19)	176 (2)

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2306).

## References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.  
 Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.  
 Danish, I. A. & Rajendra Prasad, K. J. (2004). *Indian J. Chem. Sect. B*, **43**, 618–623.  
 Danish, I. A. & Rajendra Prasad, K. J. (2005). *Collect. Czech. Chem. Commun.* **70**, 223–236.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Knölker, H. J. & Reddy, K. R. (2002). *Chem. Rev.* **102**, 4303–4428.  
 Oxford Diffraction (2010). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.  
 Periyasami, G., Raghunathan, R., Surendiran, G. & Mathivanan, N. (2008). *Bioorg. Med. Chem. Lett.* **18**, 2342–2345.  
 Periyasami, G., Raghunathan, R., Surendiran, G. & Mathivanan, N. (2009). *Eur. J. Med. Chem.* **44**, 959–966.  
 Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.  
 Spek, A. L. (2009). *Acta Cryst. D* **65**, 148–155.  
 Sridharan, M., Beagle, L. K., Zeller, M. & Rajendra Prasad, K. J. (2008). *J. Chem. Res.* pp. 572–577.

## supporting information

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**(E)-2-(Furan-2-ylmethylidene)-8-methyl-2,3,4,9-tetrahydro-1H-carbazol-1-one****R. Archana, E. Yamuna, K. J. Rajendra Prasad, A. Thiruvalluvar and R. J. Butcher****S1. Comment**

As a result of their significant potential as therapeutics, interest has grown in the development of methods for the efficient and rapid synthesis of the derivatives of various hetero-annulated carbazoles especially because the current methods, which involve multi-step reactions, lower yields, longer reaction times, and high cost of palladium (Knölker & Reddy (2002)) are unsatisfactory. Herein, therefore, we report the easily accessible synthon (*E*)-2-(furan-2-yl methylene)-8-methyl-2,3,4,9-tetrahydro-1*H*-carbazol-1-one to derive various hetero-annulated carbazoles (Sridharan *et al.*, (2008); Danish & Rajendra Prasad (2004, 2005); Periyasami *et al.*, (2008, 2009)).

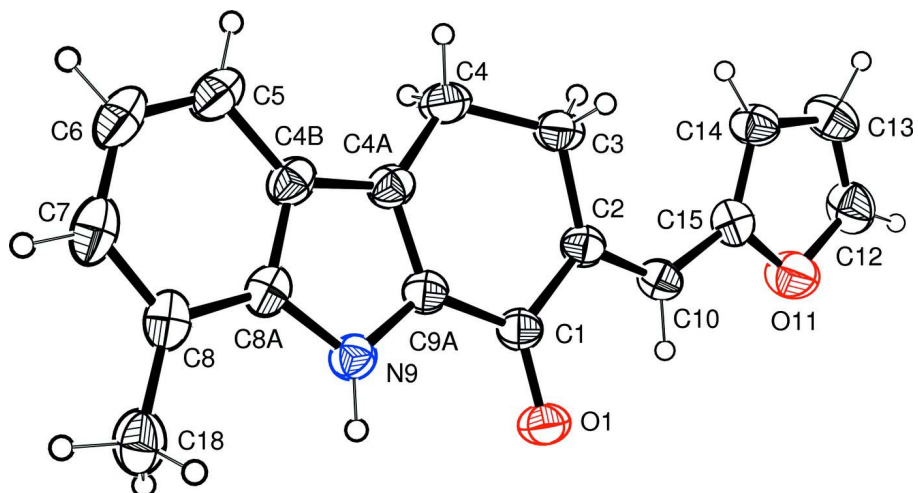
In the title molecule (Scheme I, Fig. 1), C<sub>18</sub>H<sub>15</sub>NO<sub>2</sub>, the carbazole unit is not planar. The pyrrole ring makes dihedral angles of 1.21 (10)° and 16.74 (12)° with the benzene and the furan rings respectively. The cyclohexene ring adopts a half-chair conformation. The puckering parameters (Cremer & Pople, 1975) are  $q_2=0.232$  (2) Å,  $q_3=-0.153$  (2) Å,  $Q=0.278$  (2) Å,  $\theta=123.4$  (4)° and  $\varphi=322.6$  (5)°. Intermolecular N9—H9⋯O1 hydrogen bonds form a  $R_2^2(10)$  (Bernstein *et al.*, 1995) ring in the crystal structure (Table 1, Fig. 2).

**S2. Experimental**

An equimolar mixture of 8-methyl-2,3,4,9-tetrahydro-1*H*-carbazol-1-one (0.995 g, 0.005 mol) and furan-2-carbaldehyde (0.41 ml, 0.005 mol) was treated with 25 ml of a 5% ethanolic potassium hydroxide solution and stirred for 6 h at room temperature. The product precipitated as a yellow crystalline mass, was filtered off and washed with 50% ethanol. A further crop of condensation product was obtained on neutralization with acetic acid and dilution with water. The product was recrystallized from methanol to yield 90% (1.246 g) of the title compound. The pure compound was recrystallized from EtOAc.

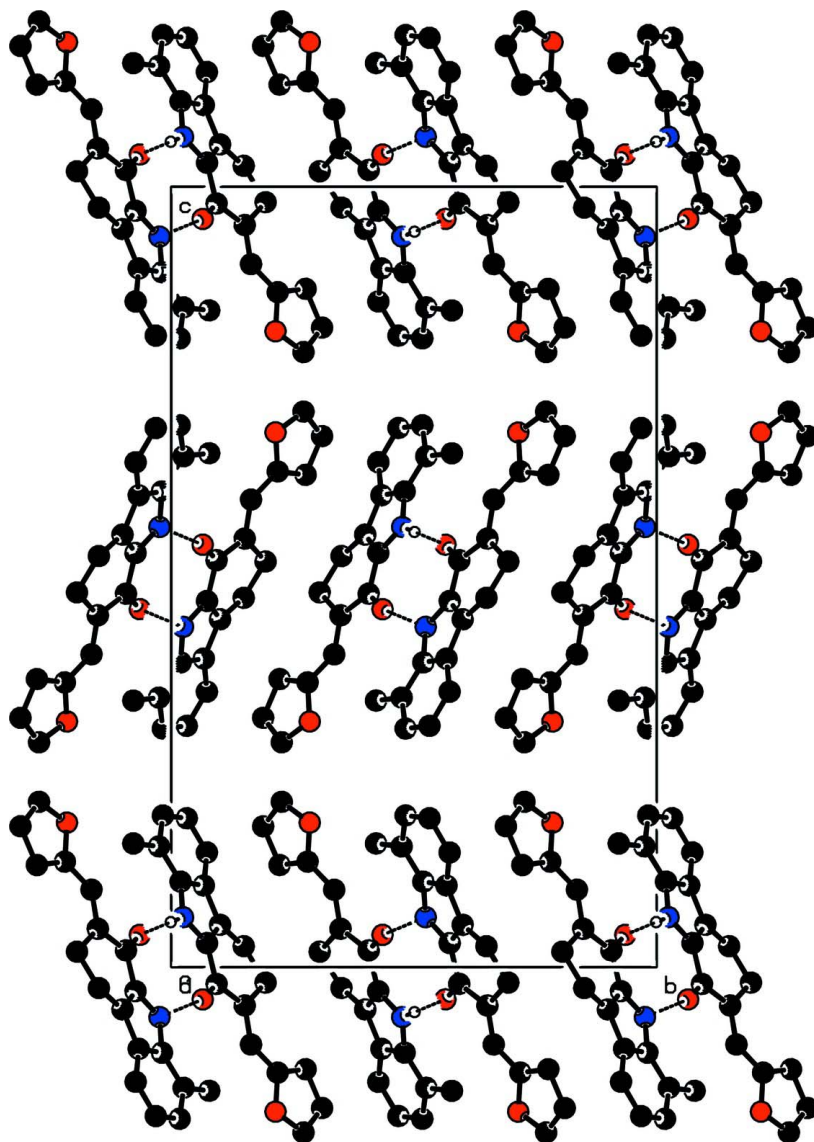
**S3. Refinement**

The H atom bonded to N9 was located in a difference Fourier map and refined freely. Other H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.97 Å and  $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{parent atom})$ .



**Figure 1**

The molecular structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level. H atoms are shown as small 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.

**(*E*)-2-(Furan-2-ylmethylidene)-8-methyl-2,3,4,9-tetrahydro-1*H*-carbazol-1-one**

*Crystal data*

$C_{18}H_{15}NO_2$

$M_r = 277.31$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 6.7353$  (1) Å

$b = 16.1393$  (3) Å

$c = 25.9549$  (4) Å

$V = 2821.38$  (8) Å<sup>3</sup>

$Z = 8$

$F(000) = 1168$

$D_x = 1.306$  Mg m<sup>-3</sup>

Melting point: 505 K

Cu *K*α radiation,  $\lambda = 1.54184$  Å

Cell parameters from 3160 reflections

$\theta = 4.4\text{--}72.5^\circ$

$\mu = 0.68$  mm<sup>-1</sup>

$T = 295$  K

Prism, pale-yellow

$0.44 \times 0.28 \times 0.12$  mm

*Data collection*

Oxford Diffraction Xcalibur Ruby Gemini  
diffractometer  
Radiation source: Enhance (Cu) X-ray Source  
Graphite monochromator  
Detector resolution: 10.5081 pixels mm<sup>-1</sup>  
 $\omega$  scans  
Absorption correction: multi-scan  
(*CrysAlis PRO*; Oxford Diffraction, 2010)  
 $T_{\min} = 0.888$ ,  $T_{\max} = 1.000$

6361 measured reflections  
2736 independent reflections  
2201 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.026$   
 $\theta_{\max} = 72.7^\circ$ ,  $\theta_{\min} = 5.7^\circ$   
 $h = -5 \rightarrow 8$   
 $k = -19 \rightarrow 13$   
 $l = -31 \rightarrow 31$

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.050$   
 $wR(F^2) = 0.157$   
 $S = 1.05$   
2736 reflections  
195 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  
 $w = 1/[\sigma^2(F_o^2) + (0.0921P)^2 + 0.408P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$

*Special details*

**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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.3691 (2)	0.06478 (9)	-0.04131 (5)	0.0638 (5)
O11	0.0871 (3)	0.21485 (11)	-0.18520 (6)	0.0810 (6)
N9	0.2678 (2)	0.02422 (9)	0.06403 (5)	0.0489 (4)
C1	0.2176 (3)	0.09332 (11)	-0.02148 (6)	0.0475 (5)
C2	0.0821 (2)	0.15134 (10)	-0.04916 (7)	0.0473 (5)
C3	-0.0829 (3)	0.19426 (12)	-0.01982 (8)	0.0605 (6)
C4	-0.1653 (3)	0.14925 (13)	0.02677 (8)	0.0615 (6)
C4A	-0.0102 (3)	0.10045 (11)	0.05422 (7)	0.0508 (5)
C4B	-0.0102 (3)	0.06700 (11)	0.10466 (7)	0.0537 (6)
C5	-0.1417 (3)	0.07341 (14)	0.14698 (8)	0.0679 (7)
C6	-0.0955 (4)	0.03192 (16)	0.19083 (9)	0.0735 (8)
C7	0.0762 (3)	-0.01716 (14)	0.19432 (8)	0.0681 (7)
C8	0.2108 (3)	-0.02535 (12)	0.15467 (7)	0.0561 (6)
C8A	0.1646 (3)	0.01922 (10)	0.10952 (6)	0.0497 (5)
C9A	0.1612 (3)	0.07363 (10)	0.03084 (6)	0.0466 (5)
C10	0.1218 (3)	0.16433 (11)	-0.09919 (7)	0.0518 (5)

C12	-0.0226 (4)	0.27239 (15)	-0.21183 (9)	0.0775 (8)
C13	-0.1434 (4)	0.31065 (15)	-0.18050 (9)	0.0736 (8)
C14	-0.1132 (3)	0.27681 (12)	-0.13083 (7)	0.0586 (6)
C15	0.0280 (3)	0.21920 (11)	-0.13515 (7)	0.0551 (6)
C18	0.3917 (3)	-0.07814 (15)	0.15847 (8)	0.0718 (8)
H3A	-0.19153	0.20433	-0.04355	0.0725*
H3B	-0.03417	0.24784	-0.00856	0.0725*
H4A	-0.22223	0.18937	0.05035	0.0738*
H4B	-0.27067	0.11228	0.01579	0.0738*
H5	-0.25654	0.10519	0.14480	0.0815*
H6	-0.17930	0.03607	0.21919	0.0882*
H7	0.10015	-0.04552	0.22489	0.0818*
H9	0.382 (4)	-0.0012 (13)	0.0577 (9)	0.063 (6)*
H10	0.22576	0.13304	-0.11252	0.0622*
H12	-0.01222	0.28261	-0.24699	0.0931*
H13	-0.23267	0.35233	-0.18921	0.0884*
H14	-0.17937	0.29187	-0.10080	0.0704*
H18A	0.39152	-0.10687	0.19085	0.1077*
H18B	0.50785	-0.04389	0.15617	0.1077*
H18C	0.39214	-0.11766	0.13083	0.1077*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0598 (8)	0.0799 (9)	0.0517 (7)	0.0239 (7)	0.0087 (6)	0.0061 (6)
O11	0.0930 (12)	0.0872 (11)	0.0627 (9)	0.0242 (9)	0.0049 (8)	0.0038 (8)
N9	0.0503 (8)	0.0537 (8)	0.0428 (7)	0.0025 (7)	0.0021 (6)	-0.0015 (6)
C1	0.0486 (9)	0.0478 (9)	0.0460 (9)	0.0025 (7)	0.0006 (7)	-0.0052 (7)
C2	0.0471 (9)	0.0464 (8)	0.0485 (9)	0.0012 (7)	-0.0018 (7)	-0.0039 (7)
C3	0.0562 (10)	0.0619 (11)	0.0633 (12)	0.0158 (9)	0.0043 (9)	0.0010 (9)
C4	0.0534 (10)	0.0664 (11)	0.0647 (12)	0.0118 (9)	0.0092 (9)	-0.0047 (9)
C4A	0.0500 (9)	0.0496 (9)	0.0528 (10)	-0.0019 (7)	0.0042 (7)	-0.0084 (8)
C4B	0.0550 (10)	0.0557 (10)	0.0503 (10)	-0.0077 (8)	0.0080 (8)	-0.0092 (8)
C5	0.0656 (12)	0.0755 (13)	0.0627 (12)	-0.0084 (10)	0.0200 (10)	-0.0104 (10)
C6	0.0797 (15)	0.0856 (15)	0.0553 (12)	-0.0232 (12)	0.0225 (11)	-0.0094 (11)
C7	0.0816 (15)	0.0771 (13)	0.0457 (10)	-0.0289 (12)	0.0024 (10)	0.0011 (9)
C8	0.0672 (11)	0.0566 (10)	0.0444 (9)	-0.0180 (9)	-0.0046 (8)	-0.0018 (8)
C8A	0.0546 (9)	0.0514 (9)	0.0431 (9)	-0.0118 (8)	0.0025 (7)	-0.0072 (7)
C9A	0.0491 (8)	0.0462 (8)	0.0446 (9)	0.0009 (7)	-0.0007 (7)	-0.0055 (7)
C10	0.0548 (9)	0.0478 (9)	0.0529 (10)	0.0049 (8)	-0.0025 (8)	-0.0045 (7)
C12	0.0962 (16)	0.0823 (14)	0.0541 (12)	0.0093 (13)	-0.0067 (12)	0.0136 (11)
C13	0.0794 (14)	0.0662 (13)	0.0753 (15)	0.0177 (11)	-0.0178 (12)	0.0001 (11)
C14	0.0643 (11)	0.0643 (11)	0.0473 (10)	0.0131 (9)	-0.0024 (8)	-0.0073 (8)
C15	0.0623 (10)	0.0526 (9)	0.0505 (10)	-0.0036 (8)	-0.0064 (8)	-0.0008 (8)
C18	0.0778 (14)	0.0819 (15)	0.0556 (12)	-0.0085 (12)	-0.0087 (10)	0.0123 (10)

*Geometric parameters (Å, °)*

O1—C1	1.232 (2)	C8—C8A	1.410 (2)
O11—C12	1.373 (3)	C10—C15	1.433 (3)
O11—C15	1.361 (2)	C12—C13	1.306 (4)
N9—C8A	1.373 (2)	C13—C14	1.415 (3)
N9—C9A	1.376 (2)	C14—C15	1.335 (3)
N9—H9	0.89 (3)	C3—H3A	0.9700
C1—C2	1.492 (2)	C3—H3B	0.9700
C1—C9A	1.446 (2)	C4—H4A	0.9700
C2—C3	1.515 (3)	C4—H4B	0.9700
C2—C10	1.342 (3)	C5—H5	0.9300
C3—C4	1.516 (3)	C6—H6	0.9300
C4—C4A	1.490 (3)	C7—H7	0.9300
C4A—C9A	1.374 (3)	C10—H10	0.9300
C4A—C4B	1.416 (3)	C12—H12	0.9300
C4B—C5	1.415 (3)	C13—H13	0.9300
C4B—C8A	1.413 (3)	C14—H14	0.9300
C5—C6	1.357 (3)	C18—H18A	0.9600
C6—C7	1.405 (3)	C18—H18B	0.9600
C7—C8	1.378 (3)	C18—H18C	0.9600
C8—C18	1.490 (3)		
O1…N9	2.8930 (19)	C14…H5 <sup>iii</sup>	3.0900
O1…N9 <sup>i</sup>	2.8969 (19)	C14…H3A	2.6000
O11…C7 <sup>ii</sup>	3.383 (3)	C15…H3A	2.8100
O1…H9	2.78 (2)	C18…H9	2.90 (2)
O1…H9 <sup>i</sup>	2.01 (3)	C18…H10 <sup>i</sup>	2.9700
O1…H10	2.3600	H3A…C14	2.6000
N9…O1	2.8930 (19)	H3A…C15	2.8100
N9…O1 <sup>i</sup>	2.8969 (19)	H3A…H14	2.0500
C1…C4A <sup>ii</sup>	3.529 (3)	H3B…C4 <sup>iii</sup>	3.0300
C2…C8A <sup>ii</sup>	3.577 (2)	H3B…H4A <sup>iii</sup>	2.5700
C3…C14	3.181 (3)	H4A…C2 <sup>iv</sup>	2.8900
C4A…C1 <sup>ii</sup>	3.529 (3)	H4A…C10 <sup>iv</sup>	2.8800
C7…O11 <sup>ii</sup>	3.383 (3)	H4A…H3B <sup>iv</sup>	2.5700
C8…C15 <sup>ii</sup>	3.554 (3)	H5…C13 <sup>iv</sup>	3.0800
C8…C10 <sup>ii</sup>	3.482 (3)	H5…C14 <sup>iv</sup>	3.0900
C8A…C10 <sup>ii</sup>	3.545 (3)	H6…C7 <sup>vii</sup>	2.9100
C8A…C2 <sup>ii</sup>	3.577 (2)	H6…H7 <sup>vii</sup>	2.4600
C8A…C13 <sup>iii</sup>	3.550 (3)	H7…H18A	2.3700
C9A…C9A <sup>ii</sup>	3.595 (2)	H7…H6 <sup>vi</sup>	2.4600
C10…C8A <sup>ii</sup>	3.545 (3)	H9…O1	2.78 (2)
C10…C8 <sup>ii</sup>	3.482 (3)	H9…C18	2.90 (2)
C13…C8A <sup>iv</sup>	3.550 (3)	H9…O1 <sup>i</sup>	2.01 (3)
C14…C3	3.181 (3)	H10…O1	2.3600
C15…C8 <sup>ii</sup>	3.554 (3)	H10…C18 <sup>i</sup>	2.9700
C2…H4A <sup>iii</sup>	2.8900	H10…H18B <sup>i</sup>	2.5600

C3...H14	2.7100	H13...C6 <sup>iv</sup>	3.0800
C4...H3B <sup>iv</sup>	3.0300	H13...C7 <sup>iv</sup>	2.9600
C4A...H14 <sup>iii</sup>	3.0700	H13...C8 <sup>iv</sup>	2.9600
C5...H18B <sup>v</sup>	3.0400	H13...C8A <sup>iv</sup>	3.0100
C6...H18B <sup>v</sup>	3.0700	H14...C3	2.7100
C6...H13 <sup>iii</sup>	3.0800	H14...H3A	2.0500
C7...H6 <sup>vi</sup>	2.9100	H14...C4A <sup>iv</sup>	3.0700
C7...H13 <sup>iii</sup>	2.9600	H14...C9A <sup>iv</sup>	3.0300
C8...H13 <sup>iii</sup>	2.9600	H18A...H7	2.3700
C8A...H13 <sup>iii</sup>	3.0100	H18B...C5 <sup>viii</sup>	3.0400
C9A...H14 <sup>iii</sup>	3.0300	H18B...C6 <sup>viii</sup>	3.0700
C10...H4A <sup>iii</sup>	2.8800	H18B...H10 <sup>i</sup>	2.5600
C13...H5 <sup>iii</sup>	3.0800		
C12—O11—C15	106.75 (18)	C10—C15—C14	133.61 (18)
C8A—N9—C9A	107.96 (14)	O11—C15—C10	117.45 (17)
C9A—N9—H9	127.2 (15)	O11—C15—C14	108.94 (17)
C8A—N9—H9	124.8 (15)	C2—C3—H3A	108.00
O1—C1—C9A	121.86 (17)	C2—C3—H3B	108.00
O1—C1—C2	122.69 (15)	C4—C3—H3A	108.00
C2—C1—C9A	115.44 (16)	C4—C3—H3B	108.00
C3—C2—C10	124.13 (16)	H3A—C3—H3B	107.00
C1—C2—C3	119.58 (16)	C3—C4—H4A	109.00
C1—C2—C10	116.23 (15)	C3—C4—H4B	109.00
C2—C3—C4	116.77 (16)	C4A—C4—H4A	109.00
C3—C4—C4A	112.22 (17)	C4A—C4—H4B	109.00
C4B—C4A—C9A	106.75 (16)	H4A—C4—H4B	108.00
C4—C4A—C4B	130.07 (18)	C4B—C5—H5	121.00
C4—C4A—C9A	122.99 (16)	C6—C5—H5	121.00
C5—C4B—C8A	119.49 (17)	C5—C6—H6	119.00
C4A—C4B—C5	133.62 (19)	C7—C6—H6	119.00
C4A—C4B—C8A	106.89 (16)	C6—C7—H7	118.00
C4B—C5—C6	118.1 (2)	C8—C7—H7	118.00
C5—C6—C7	121.4 (2)	C2—C10—H10	115.00
C6—C7—C8	123.2 (2)	C15—C10—H10	115.00
C7—C8—C8A	115.26 (18)	O11—C12—H12	125.00
C8A—C8—C18	121.82 (17)	C13—C12—H12	125.00
C7—C8—C18	122.92 (18)	C12—C13—H13	126.00
N9—C8A—C8	129.29 (17)	C14—C13—H13	126.00
C4B—C8A—C8	122.45 (17)	C13—C14—H14	126.00
N9—C8A—C4B	108.25 (14)	C15—C14—H14	126.00
C1—C9A—C4A	124.50 (17)	C8—C18—H18A	109.00
N9—C9A—C1	125.34 (17)	C8—C18—H18B	109.00
N9—C9A—C4A	110.15 (14)	C8—C18—H18C	109.00
C2—C10—C15	129.69 (17)	H18A—C18—H18B	109.00
O11—C12—C13	110.0 (2)	H18A—C18—H18C	109.00
C12—C13—C14	107.2 (2)	H18B—C18—H18C	109.00
C13—C14—C15	107.15 (18)		



C15—O11—C12—C13	0.0 (3)	C9A—C4A—C4B—C8A	-0.8 (2)
C12—O11—C15—C10	179.89 (19)	C4—C4A—C9A—N9	-174.75 (17)
C12—O11—C15—C14	0.2 (2)	C4—C4A—C9A—C1	4.0 (3)
C9A—N9—C8A—C4B	-0.28 (19)	C4B—C4A—C9A—N9	0.6 (2)
C9A—N9—C8A—C8	178.49 (18)	C4B—C4A—C9A—C1	179.32 (17)
C8A—N9—C9A—C1	-178.91 (17)	C4A—C4B—C5—C6	179.8 (2)
C8A—N9—C9A—C4A	-0.2 (2)	C8A—C4B—C5—C6	-1.0 (3)
O1—C1—C2—C3	-170.47 (17)	C4A—C4B—C8A—N9	0.7 (2)
O1—C1—C2—C10	7.0 (3)	C4A—C4B—C8A—C8	-178.22 (17)
C9A—C1—C2—C3	8.5 (2)	C5—C4B—C8A—N9	-178.77 (17)
C9A—C1—C2—C10	-174.06 (16)	C5—C4B—C8A—C8	2.4 (3)
O1—C1—C9A—N9	1.6 (3)	C4B—C5—C6—C7	-0.9 (3)
O1—C1—C9A—C4A	-176.90 (18)	C5—C6—C7—C8	1.5 (4)
C2—C1—C9A—N9	-177.39 (15)	C6—C7—C8—C8A	-0.1 (3)
C2—C1—C9A—C4A	4.1 (3)	C6—C7—C8—C18	-179.4 (2)
C1—C2—C3—C4	-28.3 (2)	C7—C8—C8A—N9	179.62 (18)
C10—C2—C3—C4	154.45 (18)	C7—C8—C8A—C4B	-1.8 (3)
C1—C2—C10—C15	-176.11 (18)	C18—C8—C8A—N9	-1.1 (3)
C3—C2—C10—C15	1.2 (3)	C18—C8—C8A—C4B	177.53 (18)
C2—C3—C4—C4A	33.9 (2)	C2—C10—C15—O11	-174.38 (19)
C3—C4—C4A—C4B	162.79 (19)	C2—C10—C15—C14	5.2 (4)
C3—C4—C4A—C9A	-23.0 (3)	O11—C12—C13—C14	-0.2 (3)
C4—C4A—C4B—C5	-6.5 (4)	C12—C13—C14—C15	0.4 (3)
C4—C4A—C4B—C8A	174.15 (19)	C13—C14—C15—O11	-0.4 (2)
C9A—C4A—C4B—C5	178.5 (2)	C13—C14—C15—C10	-180.0 (2)

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

*Hydrogen-bond geometry* ( $\text{\AA}, ^\circ$ )

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N9—H9 $\cdots$ O1 <sup>i</sup>	0.89 (3)	2.01 (3)	2.8969 (19)	176 (2)

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