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Structure Reports

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6-Chloro-3,4-dihydro-9H-carbazol-1(2H)-one

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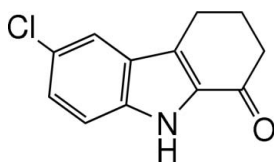
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Key indicators: single-crystal X-ray study; $T = 200$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.063; wR factor = 0.140; data-to-parameter ratio = 27.9.

The carbazole unit of the title molecule, $\text{C}_{12}\text{H}_{10}\text{ClNO}$, is not planar. The dihedral angle between the benzene and pyrrole rings is 1.35 (10)°. The cyclohexene ring adopts an envelope conformation. In the crystal structure, intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds form centrosymmetric dimers.

Related literature

For a related structure with a non-planar carbazole unit, see: Sridharan *et al.* (2008).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{10}\text{ClNO}$
 $M_r = 219.66$
 Monoclinic, $P2_1/c$
 $a = 10.4211$ (5) Å
 $b = 5.6851$ (3) Å
 $c = 17.0824$ (10) Å
 $\beta = 100.239$ (6)°

$V = 995.93$ (9) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.35$ mm⁻¹
 $T = 200$ (2) K
 $0.58 \times 0.18 \times 0.11$ mm

Data collection

Oxford Diffraction R Gemini diffractometer
 Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2008)

$T_{\min} = 0.923$, $T_{\max} = 1.000$
 (expected range = 0.888–0.962)
 10695 measured reflections
 3909 independent reflections
 1793 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.072$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.139$
 $S = 0.88$
 3909 reflections
 140 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.46$ e Å⁻³
 $\Delta\rho_{\min} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N9}-\text{H9}\cdots\text{O1}^i$	0.82 (2)	2.11 (2)	2.872 (2)	154 (2)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2008); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2008); data reduction: *CrysAlis RED*; 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: SJ2518).

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supplementary materials

Acta Cryst. (2008). E64, o1635 [doi:10.1107/S1600536808023441]

6-Chloro-3,4-dihydro-9*H*-carbazol-1(2*H*)-one

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Comment

Sridharan *et al.* (2008) have reported the crystal structure of 6-Methoxy-2,3,4,9-tetrahydro-1*H*-carbazol-1-one, in which the carbazole unit is 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 1.35 (10)°. The cyclohexene ring adopts an envelope conformation. Intermolecular N9—H9···O1 ($-x, -y, -z$) hydrogen bonds form centrosymmetric dimers in the crystal structure, Fig. 2.

Experimental

A solution of 2-(2-(4-chlorophenyl)hydrazono)cyclohexanone (0.236 g, 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 contents were then cooled and poured onto cold water with stirring. The brown solid which separated was purified by passing through a column of silica gel and eluting with (95:5 v/v) petroleum ether-ethyl acetate mixture to yield the title compound (0.153 g, 70%). This was recrystallized from ethanol.

Refinement

The crystal used was very weakly diffracting particularly at high Bragg angles. 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 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$.

Figures

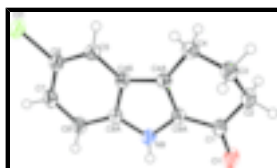


Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level. Hydrogen atoms are represented by spheres of arbitrary radius.

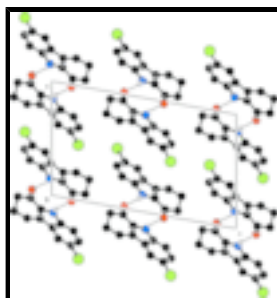


Fig. 2. The molecular packing of the title compound, viewed down the *b* axis. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

6-Chloro-3,4-dihydro-9H-carbazol-1(2H)-one

Crystal data

$C_{12}H_{10}ClNO$	$F_{000} = 456$
$M_r = 219.66$	$D_x = 1.465 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Melting point: 475(1) K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation
$a = 10.4211 (5) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 5.6851 (3) \text{ \AA}$	Cell parameters from 1948 reflections
$c = 17.0824 (10) \text{ \AA}$	$\theta = 4.6\text{--}34.7^\circ$
$\beta = 100.239 (6)^\circ$	$\mu = 0.35 \text{ mm}^{-1}$
$V = 995.93 (9) \text{ \AA}^3$	$T = 200 (2) \text{ K}$
$Z = 4$	Needle, colourless
	$0.58 \times 0.18 \times 0.11 \text{ mm}$

Data collection

Oxford Diffraction R Gemini diffractometer	3909 independent reflections
Radiation source: fine-focus sealed tube	1793 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.072$
Detector resolution: 10.5081 pixels mm^{-1}	$\theta_{\text{max}} = 34.7^\circ$
$T = 200(2) \text{ K}$	$\theta_{\text{min}} = 4.6^\circ$
φ and ω scans	$h = -16 \rightarrow 15$
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2008)	$k = -9 \rightarrow 9$
$T_{\text{min}} = 0.923$, $T_{\text{max}} = 1.000$	$l = -27 \rightarrow 19$
10695 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.062$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.139$	$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2]$
$S = 0.88$	where $P = (F_o^2 + 2F_c^2)/3$
3909 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
140 parameters	$\Delta\rho_{\text{max}} = 0.46 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
	Extinction correction: none

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.51516 (5)	0.81197 (10)	-0.14449 (3)	0.0418 (2)
O1	-0.02083 (14)	0.1402 (3)	0.10672 (9)	0.0403 (5)
N9	0.14769 (15)	0.2338 (3)	-0.00949 (10)	0.0293 (5)
C1	0.05496 (17)	0.3080 (3)	0.11410 (11)	0.0276 (5)
C2	0.06417 (19)	0.4778 (4)	0.18253 (12)	0.0340 (6)
C3	0.1973 (2)	0.5983 (4)	0.20336 (12)	0.0361 (6)
C4	0.2388 (2)	0.7173 (4)	0.13273 (12)	0.0358 (7)
C4A	0.22083 (17)	0.5536 (3)	0.06315 (11)	0.0258 (5)
C4B	0.28050 (17)	0.5475 (3)	-0.00559 (11)	0.0251 (5)
C5	0.36953 (17)	0.6955 (3)	-0.03472 (12)	0.0286 (5)
C6	0.40706 (18)	0.6321 (3)	-0.10471 (12)	0.0292 (6)
C7	0.36271 (19)	0.4274 (4)	-0.14660 (12)	0.0337 (6)
C8	0.27603 (19)	0.2811 (4)	-0.11885 (12)	0.0318 (6)
C8A	0.23419 (17)	0.3435 (3)	-0.04868 (11)	0.0260 (5)
C9A	0.13871 (17)	0.3614 (3)	0.05784 (11)	0.0253 (5)
H2A	-0.00399	0.59971	0.16903	0.0408*
H2B	0.04640	0.39204	0.22991	0.0408*
H3A	0.26364	0.47952	0.22511	0.0433*
H3B	0.19391	0.71695	0.24535	0.0433*
H4A	0.33155	0.76429	0.14653	0.0430*
H4B	0.18603	0.86099	0.11876	0.0430*
H5	0.40231	0.83389	-0.00698	0.0343*
H7	0.39275	0.38971	-0.19443	0.0404*
H8	0.24555	0.14153	-0.14666	0.0381*
H9	0.1137 (19)	0.107 (4)	-0.0230 (12)	0.023 (5)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0407 (3)	0.0437 (3)	0.0464 (3)	-0.0017 (2)	0.0224 (2)	0.0095 (3)
O1	0.0350 (7)	0.0496 (9)	0.0389 (8)	-0.0151 (7)	0.0138 (7)	-0.0069 (8)
N9	0.0308 (8)	0.0319 (9)	0.0269 (8)	-0.0096 (7)	0.0095 (7)	-0.0062 (8)
C1	0.0222 (8)	0.0333 (10)	0.0273 (9)	0.0006 (8)	0.0046 (7)	0.0012 (9)

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C2	0.0336 (10)	0.0437 (12)	0.0271 (10)	-0.0017 (9)	0.0119 (9)	-0.0023 (10)
C3	0.0424 (11)	0.0397 (12)	0.0279 (10)	-0.0077 (9)	0.0109 (9)	-0.0051 (10)
C4	0.0459 (12)	0.0306 (11)	0.0333 (11)	-0.0057 (9)	0.0138 (10)	-0.0061 (10)
C4A	0.0265 (9)	0.0262 (9)	0.0250 (9)	0.0022 (7)	0.0051 (8)	0.0012 (9)
C4B	0.0255 (8)	0.0275 (10)	0.0223 (9)	0.0020 (7)	0.0043 (7)	0.0035 (8)
C5	0.0302 (9)	0.0269 (10)	0.0294 (9)	-0.0009 (8)	0.0071 (8)	0.0023 (9)
C6	0.0273 (9)	0.0319 (11)	0.0305 (10)	0.0040 (7)	0.0113 (8)	0.0085 (9)
C7	0.0355 (10)	0.0412 (12)	0.0266 (9)	0.0051 (9)	0.0117 (9)	0.0005 (10)
C8	0.0344 (10)	0.0348 (11)	0.0276 (10)	-0.0007 (8)	0.0097 (8)	-0.0046 (9)
C8A	0.0244 (8)	0.0302 (10)	0.0240 (9)	0.0004 (7)	0.0057 (7)	0.0006 (9)
C9A	0.0240 (8)	0.0287 (10)	0.0238 (9)	0.0026 (7)	0.0056 (7)	0.0007 (8)

Geometric parameters (Å, °)

C11—C6	1.7460 (19)	C5—C6	1.371 (3)
O1—C1	1.231 (2)	C6—C7	1.401 (3)
N9—C8A	1.365 (2)	C7—C8	1.373 (3)
N9—C9A	1.377 (2)	C8—C8A	1.392 (3)
N9—H9	0.82 (2)	C2—H2A	0.9900
C1—C9A	1.441 (3)	C2—H2B	0.9900
C1—C2	1.506 (3)	C3—H3A	0.9900
C2—C3	1.531 (3)	C3—H3B	0.9900
C3—C4	1.512 (3)	C4—H4A	0.9900
C4—C4A	1.495 (3)	C4—H4B	0.9900
C4A—C4B	1.424 (3)	C5—H5	0.9500
C4A—C9A	1.381 (2)	C7—H7	0.9500
C4B—C8A	1.412 (2)	C8—H8	0.9500
C4B—C5	1.407 (3)		
C11...C4A ⁱ	3.5299 (19)	C8A...H2A ^{vi}	2.8900
C11...H7 ⁱⁱ	3.1000	C9A...H3A	3.0000
C11...H4A ⁱⁱⁱ	2.8900	C9A...H4B ^v	3.0400
O1...N9	2.924 (2)	H2A...H2B ^x	2.4900
O1...N9 ^{iv}	2.872 (2)	H2A...C8 ^{vi}	2.8900
O1...H4B ^v	2.6600	H2A...C8A ^{vi}	2.8900
O1...H9	2.83 (2)	H2B...H2A ^{xi}	2.4900
O1...H9 ^{iv}	2.11 (2)	H3A...C9A	3.0000
N9...O1	2.924 (2)	H3A...C8 ^{vii}	3.0300
N9...O1 ^{iv}	2.872 (2)	H3A...H8 ^{vii}	2.3400
C4A...C11 ⁱ	3.5299 (19)	H3B...C7 ^{xii}	3.0700
C5...C5 ⁱ	3.549 (3)	H4A...C11 ⁱⁱⁱ	2.8900
C5...C6 ⁱ	3.548 (3)	H4B...O1 ^{xiii}	2.6600
C6...C5 ⁱ	3.548 (3)	H4B...C1 ^{xiii}	2.8800
C9A...C9A ^{vi}	3.566 (3)	H4B...C9A ^{xiii}	3.0400
C1...H4B ^v	2.8800	H7...C11 ^{xiv}	3.1000
C3...H8 ^{vii}	2.8700	H8...C3 ^{ix}	2.8700

C7...H3B ^{viii}	3.0700	H8...H3A ^{ix}	2.3400
C8...H2A ^{vi}	2.8900	H9...O1	2.83 (2)
C8...H3A ^{ix}	3.0300	H9...O1 ^{iv}	2.11 (2)
C8A—N9—C9A	108.53 (15)	C1—C9A—C4A	124.38 (17)
C9A—N9—H9	127.6 (14)	N9—C9A—C1	125.83 (16)
C8A—N9—H9	123.7 (14)	N9—C9A—C4A	109.78 (16)
O1—C1—C9A	123.29 (17)	C1—C2—H2A	109.00
O1—C1—C2	121.85 (17)	C1—C2—H2B	109.00
C2—C1—C9A	114.85 (16)	C3—C2—H2A	109.00
C1—C2—C3	113.41 (16)	C3—C2—H2B	109.00
C2—C3—C4	112.99 (17)	H2A—C2—H2B	108.00
C3—C4—C4A	110.04 (18)	C2—C3—H3A	109.00
C4B—C4A—C9A	106.44 (16)	C2—C3—H3B	109.00
C4—C4A—C4B	131.28 (17)	C4—C3—H3A	109.00
C4—C4A—C9A	122.24 (17)	C4—C3—H3B	109.00
C5—C4B—C8A	119.51 (17)	H3A—C3—H3B	108.00
C4A—C4B—C8A	106.93 (15)	C3—C4—H4A	110.00
C4A—C4B—C5	133.57 (17)	C3—C4—H4B	110.00
C4B—C5—C6	117.36 (16)	C4A—C4—H4A	110.00
C11—C6—C5	119.30 (14)	C4A—C4—H4B	110.00
C11—C6—C7	117.70 (15)	H4A—C4—H4B	108.00
C5—C6—C7	123.00 (17)	C4B—C5—H5	121.00
C6—C7—C8	120.30 (19)	C6—C5—H5	121.00
C7—C8—C8A	117.96 (19)	C6—C7—H7	120.00
N9—C8A—C8	129.85 (17)	C8—C7—H7	120.00
C4B—C8A—C8	121.86 (17)	C7—C8—H8	121.00
N9—C8A—C4B	108.29 (16)	C8A—C8—H8	121.00
C9A—N9—C8A—C4B	-0.6 (2)	C4—C4A—C4B—C8A	175.46 (19)
C9A—N9—C8A—C8	179.52 (19)	C9A—C4A—C4B—C5	178.3 (2)
C8A—N9—C9A—C1	178.12 (17)	C4B—C4A—C9A—N9	1.7 (2)
C8A—N9—C9A—C4A	-0.7 (2)	C4B—C4A—C9A—C1	-177.15 (17)
C2—C1—C9A—N9	-179.78 (17)	C4A—C4B—C8A—N9	1.6 (2)
C2—C1—C9A—C4A	-1.1 (3)	C5—C4B—C8A—C8	1.3 (3)
O1—C1—C2—C3	153.79 (19)	C4A—C4B—C8A—C8	-178.47 (18)
C9A—C1—C2—C3	-27.7 (2)	C5—C4B—C8A—N9	-178.63 (16)
O1—C1—C9A—N9	-1.3 (3)	C4A—C4B—C5—C6	179.7 (2)
O1—C1—C9A—C4A	177.38 (18)	C8A—C4B—C5—C6	0.0 (3)
C1—C2—C3—C4	53.7 (2)	C4B—C5—C6—C11	178.90 (14)
C2—C3—C4—C4A	-48.1 (2)	C4B—C5—C6—C7	-1.2 (3)
C3—C4—C4A—C4B	-157.05 (19)	C11—C6—C7—C8	-178.99 (16)
C3—C4—C4A—C9A	20.1 (3)	C5—C6—C7—C8	1.1 (3)
C4—C4A—C9A—N9	-176.05 (17)	C6—C7—C8—C8A	0.2 (3)
C4—C4A—C9A—C1	5.1 (3)	C7—C8—C8A—N9	178.52 (19)
C9A—C4A—C4B—C8A	-2.0 (2)	C7—C8—C8A—C4B	-1.4 (3)
C4—C4A—C4B—C5	-4.2 (4)		

Symmetry codes: (i) $-x+1, -y+1, -z$; (ii) $-x+1, y+1/2, -z-1/2$; (iii) $-x+1, -y+2, -z$; (iv) $-x, -y, -z$; (v) $x, y-1, z$; (vi) $-x, -y+1, -z$; (vii) $x, -y+1/2, z+1/2$; (viii) $x, -y+3/2, z-1/2$; (ix) $x, -y+1/2, z-1/2$; (x) $-x, y+1/2, -z+1/2$; (xi) $-x, y-1/2, -z+1/2$; (xii) $x, -y+3/2, z+1/2$; (xiii) $x, y+1, z$; (xiv) $-x+1, y-1/2, -z-1/2$.

supplementary materials

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N9—H9 \cdots O1 ^{iv}	0.82 (2)	2.11 (2)	2.872 (2)	154 (2)

Symmetry codes: (iv) $-x, -y, -z$.

Fig. 1

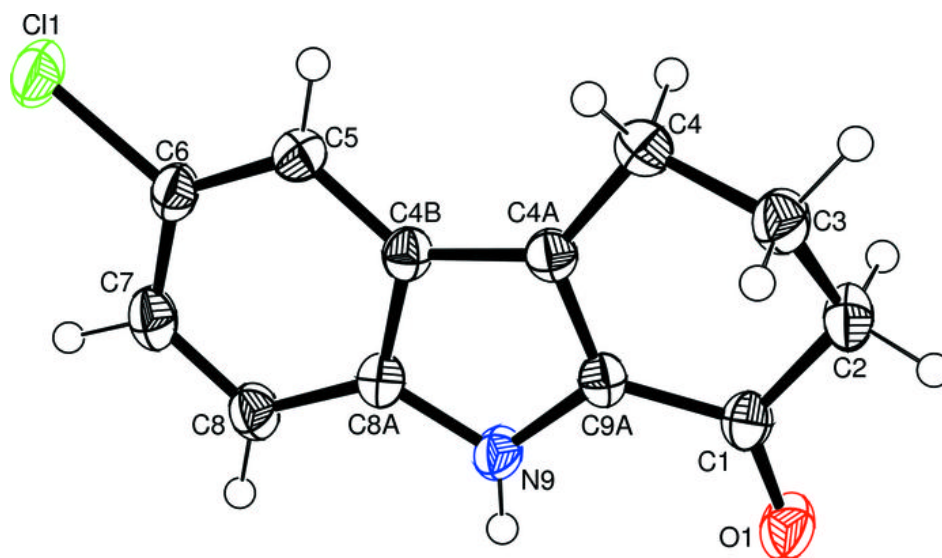


Fig. 2

