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Methyl 6-methyl-1-oxo-2,3,4,9-tetrahydrocarbazole-2-carboxylate

A. Thomas Gunaseelan,[a] A. Thiruvalluvar,[a]* A. Ebenezer Martin[b] and K. J. Rajendra Prasad[b]

[a]PG Research Department of Physics, Rajah Serfoji Government College (Autonomous), Thanjavur 613 005, Tamilnadu, India, and [b]Department of Chemistry, Bharathiyar University, Coimbatore 641 046, Tamilnadu, India
Correspondence e-mail: athiru@vsnl.net

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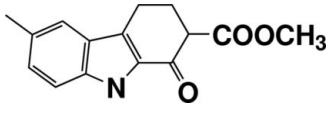
Key indicators: single-crystal X-ray study; T = 160 K; mean σ(C–C) = 0.005 Å; R factor = 0.059; wR factor = 0.162; data-to-parameter ratio = 12.5.

The carbazole unit of the title molecule, $C_{15}H_{15}NO_3$, is not planar. The dihedral angle between the benzene ring and the fused pyrrole ring is 0.53 (2)°. The cyclohexene ring is in half-chair form. The methyl acetate group at position 2 has a equatorial orientation. In the crystal structure, the molecules are stabilized by intermolecular N—H···O and C—H···O hydrogen bonds.

Related literature

The carbazole alkaloids in particular show definite antitumor characteristics (Borek-Dohalska *et al.*, 2004; Hagg *et al.*, 2004) and anti-HIV properties (Wang *et al.*, 2005) and have good prospects for future medicinal use. However, introducing a functional group into the carbazole skeleton is a tedious operation because of several side reactions (Sekar *et al.*, 1994). Here we report the crystal structure of methyl 6-methyl-1-oxo-2,3,4,9-tetrahydrocarbazole-2-carboxylate, (I), which was obtained from the decarbonylation reaction of methyl 2-(6-methyl-1-oxo-2,3,4,9-tetrahydro-1*H*-carbazol-2-yl)-2-oxoacetate.



Experimental

Crystal data

$C_{15}H_{15}NO_3$
M_r = 257.28
Triclinic, $P\overline{1}$
a = 4.6362 (6) Å
b = 10.7289 (14) Å
c = 13.4557 (18) Å
α = 69.665 (7)°
β = 88.411 (9)°
γ = 88.287 (8)°
V = 627.20 (15) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 160 (2) K
0.23 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
2228 measured reflections
2228 independent reflections
1336 reflections with $I > 2\sigma(I)$

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.162
S = 1.04
2228 reflections
178 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.231 (4)	O22—C21	1.339 (4)
O21—C21	1.206 (4)	O22—C22	1.455 (4)
C21—O22—C22	115.5 (3)	O21—C21—O22	123.3 (3)
C1—C2—C3—C4	56.2 (3)	C22—O22—C21—C2	174.3 (3)
C4—C4*A*—C9*A*—C1	2.5 (5)	C1—C2—C21—O22	−98.3 (3)
C22—O22—C21—O21	−4.6 (5)	C3—C2—C21—O22	138.2 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N9—H9···O1^{i}	0.86 (3)	2.03 (3)	2.853 (4)	158 (4)
C2—H2···O1^{ii}	1.00	2.58	3.482 (4)	150

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

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, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

The data collection was carried out by Dr A. Linden of the Institute of Organic Chemistry at the University of Zurich. This help is gratefully acknowledged by AT. AT thanks the UGC, India, for the award of a minor research project [file No. MRP-2355/06(UGC-SERO), link No. 2355, 10/01/2007].

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2271).

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supplementary materials

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Methyl 6-methyl-1-oxo-2,3,4,9-tetrahydrocarbazole-2-carboxylate

A. T. Gunaseelan, A. Thiruvalluvar, A. E. Martin and K. J. R. Prasad

Comment

The molecular structure of the title compound, (I), with atomic numbering scheme, is shown in Fig. 1. The carbazole unit is not planar. The dihedral angle between the benzene ring and the fused pyrrole ring is 0.53 (2)°. This angle confirms that the indole ring system is essentially planar. But, the cyclohexene ring is in half-chair form. The displacements of C1, C2, C4, C4A, C9A and C3 from the least-squares plane defined by atoms C1/C2/C4/C4A/C9A are -0.065 (2), 0.058 (2), -0.053 (2), 0.047 (2), 0.014 (2) and -0.608 (4) Å, respectively. This confirms that the cyclohexene ring adopts a half-chair conformation (see Table 1 for torsion angles). The methylacetate group at position 2 has a equatorial orientation. In the crystal structure, the molecules are stabilized by intermolecular N—H···O and C—H···O hydrogen bonds (see Table 2).

Experimental

A mixture of methyl 2-(6-methyl-1-oxo-2,3,4,9-tetrahydro-1*H*- carbazol-2-yl)-2-oxoacetate (285 mg, 0.001 mol), glass powder (500 mg) and iron powder (500 mg) in diphenyl ether was heated at 443 K for 30 min. The carbon monoxide will be liberated during heating. After cooling, the mixture was extracted with ethyl acetate and purified by column chromatography over silica gel using petroleum ether-ethyl acetate (95:5 v/v) as eluant (77 mg, yield 30%) and recrystallized from glacial acetic acid.

Refinement

H atom bonded to N was located in a difference map and refined freely. Other H atoms were positioned geometrically and allowed to ride on their parent atoms, with C–H = 0.95–1.00 Å and U_{iso}(H) = 1.2–1.5 times U_{eq}(C).

Figures

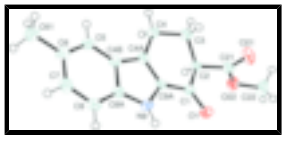

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

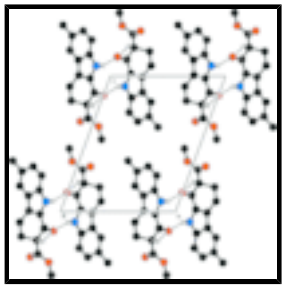

Fig. 2. The packing of (I), viewed down the a axis. Dashed lines indicate hydrogen bonds.

Methyl 6-methyl-1-oxo-2,3,4,9-tetrahydrocarbazole-2-carboxylate

Crystal data

$C_{15}H_{15}NO_3$	$Z = 2$
$M_r = 257.28$	$F_{000} = 272$
Triclinic, $P\overline{1}$	$D_x = 1.362$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 393 - 395 K
$a = 4.6362$ (6) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 10.7289$ (14) Å	Cell parameters from 2155 reflections
$c = 13.4557$ (18) Å	$\theta = 2.0$–25.0º
$\alpha = 69.665$ (7)º	$\mu = 0.10$ mm^{-1}
$\beta = 88.411$ (9)º	$T = 160$ (2) K
$\gamma = 88.287$ (8)º	Blocks, colorless
$V = 627.20$ (15) Å^3	0.23 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer	2228 independent reflections
Radiation source: Nonius FR590 sealed tube generator	1336 reflections with $I > 2\sigma(I)$
Monochromator: horizontally mounted graphite crystal	$R_{int} = 0.0000$
Detector resolution: 9 pixels mm^{-1}	$\theta_{max} = 25.1$º
$T = 160(1)$ K	$\theta_{min} = 2.0$º
ω scans with κ offsets	$h = 0 \rightarrow 5$
Absorption correction: none	$k = -12 \rightarrow 12$
2228 measured reflections	$l = -15 \rightarrow 16$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2 + 0.2914P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.059$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.162$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.24$ e Å^{-3}
2228 reflections	Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
178 parameters	Extinction coefficient: 0.028 (6)
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Hydrogen site location: inferred from neighbouring sites

Special details

Experimental. Solvent used: Cooling Device: Oxford Cryosystems Cryostream 700 Crystal mount: glued on a glass fibre Mosaicity (deg.): 1.065 (7) Frames collected: 206 Seconds exposure per frame: 38 Degrees rotation per frame: 1.9 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 esds 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.8693 (4)	−0.0008 (2)	0.14199 (17)	0.0316 (8)
O21	0.8637 (5)	0.1167 (2)	0.33351 (18)	0.0413 (9)
O22	0.5802 (5)	−0.0600 (2)	0.37179 (17)	0.0363 (8)
N9	0.7112 (6)	0.1572 (3)	−0.0758 (2)	0.0271 (10)
C1	0.6812 (6)	0.0874 (3)	0.1216 (2)	0.0236 (11)
C2	0.5031 (6)	0.1138 (3)	0.2098 (2)	0.0248 (10)
C3	0.4273 (7)	0.2617 (3)	0.1820 (3)	0.0296 (11)
C4	0.2679 (7)	0.3198 (3)	0.0771 (2)	0.0275 (11)
C4A	0.4122 (6)	0.2768 (3)	−0.0067 (2)	0.0254 (11)
C4B	0.3933 (6)	0.3303 (3)	−0.1192 (2)	0.0242 (10)
C5	0.2348 (6)	0.4353 (3)	−0.1907 (3)	0.0282 (11)
C6	0.2666 (7)	0.4629 (3)	−0.2978 (3)	0.0307 (11)
C7	0.4589 (7)	0.3828 (3)	−0.3344 (3)	0.0351 (12)
C8	0.6188 (7)	0.2792 (3)	−0.2674 (3)	0.0321 (11)
C8A	0.5847 (6)	0.2533 (3)	−0.1595 (3)	0.0250 (10)
C9A	0.6046 (6)	0.1710 (3)	0.0167 (2)	0.0233 (10)
C21	0.6692 (7)	0.0604 (3)	0.3103 (3)	0.0286 (11)
C22	0.7487 (8)	−0.1248 (4)	0.4662 (3)	0.0491 (14)
C61	0.1023 (8)	0.5762 (3)	−0.3761 (3)	0.0406 (12)
H2	0.31937	0.06400	0.21987	0.0297*
H3A	0.30498	0.27380	0.23955	0.0354*
H3B	0.60703	0.31122	0.17719	0.0354*
H4A	0.26477	0.41811	0.05404	0.0330*
H4B	0.06584	0.28989	0.08754	0.0330*
H5	0.10490	0.48756	−0.16478	0.0336*
H7	0.47854	0.40153	−0.40856	0.0422*
H8	0.74760	0.22732	−0.29406	0.0383*
H9	0.841 (7)	0.099 (3)	−0.078 (3)	0.045 (11)*
H22A	0.66743	−0.21170	0.50578	0.0739*

H22B	0.94902	−0.13677	0.44538	0.0739*
H22C	0.74332	−0.06955	0.51089	0.0739*
H61A	0.20411	0.65925	−0.38876	0.0603*
H61B	0.08768	0.55972	−0.44297	0.0603*
H61C	−0.09170	0.58344	−0.34752	0.0603*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0291 (13)	0.0345 (14)	0.0348 (14)	0.0083 (11)	−0.0026 (10)	−0.0170 (11)
O21	0.0381 (15)	0.0528 (16)	0.0353 (15)	−0.0106 (12)	−0.0063 (11)	−0.0172 (12)
O22	0.0393 (14)	0.0340 (14)	0.0328 (14)	0.0007 (11)	−0.0078 (11)	−0.0078 (12)
N9	0.0256 (16)	0.0283 (16)	0.0317 (18)	0.0069 (13)	−0.0029 (13)	−0.0162 (14)
C1	0.0185 (17)	0.0262 (18)	0.029 (2)	−0.0008 (14)	−0.0006 (14)	−0.0133 (15)
C2	0.0201 (16)	0.0328 (18)	0.0257 (19)	0.0004 (14)	0.0005 (13)	−0.0158 (15)
C3	0.0248 (18)	0.036 (2)	0.033 (2)	0.0041 (15)	0.0038 (15)	−0.0190 (16)
C4	0.0254 (17)	0.0269 (18)	0.031 (2)	0.0026 (14)	0.0050 (14)	−0.0118 (15)
C4A	0.0212 (17)	0.0262 (18)	0.031 (2)	−0.0016 (14)	0.0019 (14)	−0.0128 (15)
C4B	0.0216 (17)	0.0245 (17)	0.029 (2)	−0.0016 (14)	0.0013 (14)	−0.0124 (15)
C5	0.0249 (18)	0.0258 (18)	0.036 (2)	0.0020 (14)	0.0004 (15)	−0.0138 (16)
C6	0.0324 (19)	0.0257 (18)	0.034 (2)	0.0006 (15)	0.0004 (16)	−0.0105 (16)
C7	0.044 (2)	0.037 (2)	0.024 (2)	−0.0022 (17)	0.0029 (16)	−0.0102 (17)
C8	0.038 (2)	0.0300 (19)	0.033 (2)	0.0000 (16)	0.0037 (16)	−0.0174 (17)
C8A	0.0231 (17)	0.0241 (17)	0.029 (2)	−0.0020 (14)	−0.0012 (14)	−0.0104 (15)
C9A	0.0214 (17)	0.0285 (18)	0.0247 (19)	0.0000 (14)	−0.0002 (13)	−0.0151 (15)
C21	0.0264 (19)	0.033 (2)	0.030 (2)	0.0025 (15)	0.0028 (15)	−0.0161 (17)
C22	0.056 (3)	0.047 (2)	0.037 (2)	0.0063 (19)	−0.0137 (19)	−0.0049 (19)
C61	0.050 (2)	0.030 (2)	0.037 (2)	0.0047 (17)	−0.0021 (18)	−0.0059 (17)

Geometric parameters (Å, °)

O1—C1	1.231 (4)	C6—C7	1.414 (5)
O21—C21	1.206 (4)	C6—C61	1.506 (5)
O22—C21	1.339 (4)	C7—C8	1.375 (5)
O22—C22	1.455 (4)	C8—C8A	1.386 (5)
N9—C8A	1.367 (5)	C2—H2	1.0000
N9—C9A	1.380 (4)	C3—H3A	0.9900
N9—H9	0.86 (3)	C3—H3B	0.9900
C1—C9A	1.432 (4)	C4—H4A	0.9900
C1—C2	1.528 (4)	C4—H4B	0.9900
C2—C21	1.498 (4)	C5—H5	0.9500
C2—C3	1.531 (5)	C7—H7	0.9500
C3—C4	1.532 (5)	C8—H8	0.9500
C4—C4A	1.494 (4)	C22—H22A	0.9800
C4A—C4B	1.425 (4)	C22—H22B	0.9800
C4A—C9A	1.375 (4)	C22—H22C	0.9800
C4B—C8A	1.416 (5)	C61—H61A	0.9800
C4B—C5	1.404 (4)	C61—H61B	0.9800
C5—C6	1.371 (5)	C61—H61C	0.9800

O1···O21	3.238 (3)	C1···H4B^{iv}	2.7600
O1···O22	3.196 (3)	C5···H3B^{v}	2.9000
O1···N9	2.931 (3)	C7···H61C^{iv}	3.0000
O1···C8A^{i}	3.420 (4)	C9A···H3B	3.0300
O1···N9^{ii}	2.853 (4)	C9A···H4B^{iv}	2.8700
O21···C22^{iii}	3.308 (4)	C21···H22C^{vi}	3.0600
O21···C3^{iv}	3.339 (4)	C61···H22A^{ix}	3.0300
O21···O1	3.238 (3)	H2···O1^{vii}	2.5800
O21···C2^{iv}	3.364 (4)	H2···O21^{vii}	2.7300
O22···O1	3.196 (3)	H3A···O21^{vii}	2.6800
O22···C8^{i}	3.294 (4)	H3B···O21	2.6700
O1···H9	2.78 (4)	H3B···C9A	3.0300
O1···H9^{ii}	2.03 (3)	H3B···H4B^{iv}	2.4600
O1···H2^{iv}	2.5800	H3B···C5^{v}	2.9000
O21···H2^{iv}	2.7300	H3B···H5^{v}	2.5300
O21···H22B	2.6300	H4B···C1^{vii}	2.7600
O21···H22C	2.5800	H4B···C9A^{vii}	2.8700
O21···H22C^{iii}	2.7200	H4B···H3B^{vii}	2.4600
O21···H61A^{v}	2.7600	H5···H61C	2.5000
O21···H3A^{iv}	2.6800	H5···H3B^{v}	2.5300
O21···H3B	2.6700	H7···H61B	2.3900
O22···H8^{i}	2.8600	H8···O22^{i}	2.8600
O22···H22C^{vi}	2.8200	H9···O1	2.78 (4)
N9···O1	2.931 (3)	H9···O1^{ii}	2.03 (3)
N9···O1^{ii}	2.853 (4)	H22A···C61^{viii}	3.0300
C2···O21^{vii}	3.364 (4)	H22B···O21	2.6300
C3···O21^{vii}	3.339 (4)	H22C···O21	2.5800
C5···C8A^{vii}	3.576 (4)	H22C···O21^{iii}	2.7200
C6···C8^{vii}	3.577 (5)	H22C···O22^{vi}	2.8200
C8···C6^{iv}	3.577 (5)	H22C···C21^{vi}	3.0600
C8···O22^{i}	3.294 (4)	H61A···O21^{v}	2.7600
C8A···O1^{i}	3.420 (4)	H61B···H7	2.3900
C8A···C5^{iv}	3.576 (4)	H61B···H61B^{x}	2.4800
C22···O21^{iii}	3.308 (4)	H61C···C7^{vii}	3.0000
C22···C61^{viii}	3.554 (6)	H61C···H5	2.5000
C61···C22^{ix}	3.554 (6)		
C21—O22—C22	115.5 (3)	O21—C21—C2	124.8 (3)
C8A—N9—C9A	108.4 (3)	C1—C2—H2	108.00
C9A—N9—H9	124 (3)	C3—C2—H2	108.00
C8A—N9—H9	128 (3)	C21—C2—H2	108.00
C2—C1—C9A	114.3 (3)	C2—C3—H3A	109.00
O1—C1—C9A	124.5 (3)	C2—C3—H3B	109.00

O1—C1—C2	121.1 (2)	C4—C3—H3A	109.00
C1—C2—C3	111.8 (2)	C4—C3—H3B	109.00
C1—C2—C21	108.3 (2)	H3A—C3—H3B	108.00
C3—C2—C21	111.6 (3)	C3—C4—H4A	109.00
C2—C3—C4	112.5 (3)	C3—C4—H4B	109.00
C3—C4—C4A	110.8 (3)	C4A—C4—H4A	109.00
C4—C4A—C9A	122.5 (2)	C4A—C4—H4B	110.00
C4B—C4A—C9A	106.8 (3)	H4A—C4—H4B	108.00
C4—C4A—C4B	130.7 (3)	C4B—C5—H5	120.00
C5—C4B—C8A	118.9 (3)	C6—C5—H5	120.00
C4A—C4B—C5	134.5 (3)	C6—C7—H7	119.00
C4A—C4B—C8A	106.6 (3)	C8—C7—H7	119.00
C4B—C5—C6	120.3 (3)	C7—C8—H8	121.00
C7—C6—C61	119.9 (3)	C8A—C8—H8	121.00
C5—C6—C61	121.3 (3)	O22—C22—H22A	109.00
C5—C6—C7	118.8 (3)	O22—C22—H22B	109.00
C6—C7—C8	122.9 (3)	O22—C22—H22C	109.00
C7—C8—C8A	117.5 (3)	H22A—C22—H22B	109.00
N9—C8A—C8	130.1 (3)	H22A—C22—H22C	110.00
N9—C8A—C4B	108.4 (3)	H22B—C22—H22C	109.00
C4B—C8A—C8	121.6 (3)	C6—C61—H61A	109.00
N9—C9A—C4A	109.9 (2)	C6—C61—H61B	109.00
C1—C9A—C4A	124.9 (3)	C6—C61—H61C	109.00
N9—C9A—C1	125.3 (3)	H61A—C61—H61B	109.00
O22—C21—C2	111.9 (3)	H61A—C61—H61C	109.00
O21—C21—O22	123.3 (3)	H61B—C61—H61C	109.00
C1—C2—C3—C4	56.2 (3)	C3—C2—C21—O22	138.2 (3)
C2—C3—C4—C4A	−45.8 (4)	C3—C4—C4A—C4B	−161.4 (3)
C3—C4—C4A—C9A	17.3 (4)	C4—C4A—C4B—C5	−2.2 (6)
C4—C4A—C9A—C1	2.5 (5)	C4—C4A—C4B—C8A	177.8 (3)
C4A—C9A—C1—C2	7.0 (4)	C9A—C4A—C4B—C5	179.0 (3)
C9A—C1—C2—C3	−35.7 (4)	C9A—C4A—C4B—C8A	−1.0 (3)
C22—O22—C21—O21	−4.6 (5)	C4—C4A—C9A—N9	−177.8 (3)
C22—O22—C21—C2	174.3 (3)	C4B—C4A—C9A—N9	1.1 (4)
C9A—N9—C8A—C4B	0.1 (4)	C4B—C4A—C9A—C1	−178.6 (3)
C9A—N9—C8A—C8	−179.2 (3)	C4A—C4B—C5—C6	179.7 (3)
C8A—N9—C9A—C1	178.9 (3)	C8A—C4B—C5—C6	−0.3 (5)
C8A—N9—C9A—C4A	−0.8 (4)	C4A—C4B—C8A—N9	0.6 (4)
O1—C1—C2—C3	145.6 (3)	C4A—C4B—C8A—C8	180.0 (3)
O1—C1—C2—C21	22.2 (4)	C5—C4B—C8A—N9	−179.4 (3)
C9A—C1—C2—C21	−159.0 (3)	C5—C4B—C8A—C8	0.0 (5)
O1—C1—C9A—N9	6.1 (5)	C4B—C5—C6—C7	0.7 (5)
O1—C1—C9A—C4A	−174.3 (3)	C4B—C5—C6—C61	−179.1 (3)
C2—C1—C9A—N9	−172.6 (3)	C5—C6—C7—C8	−0.9 (5)
C21—C2—C3—C4	177.7 (3)	C61—C6—C7—C8	179.0 (3)
C1—C2—C21—O21	80.5 (4)	C6—C7—C8—C8A	0.5 (5)
C1—C2—C21—O22	−98.3 (3)	C7—C8—C8A—N9	179.2 (3)
C3—C2—C21—O21	−43.0 (4)	C7—C8—C8A—C4B	−0.1 (5)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N9—H9···O1^{ii}	0.86 (3)	2.03 (3)	2.853 (4)	158 (4)
C2—H2···O1^{vii}	1.00	2.58	3.482 (4)	150

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

Fig. 1

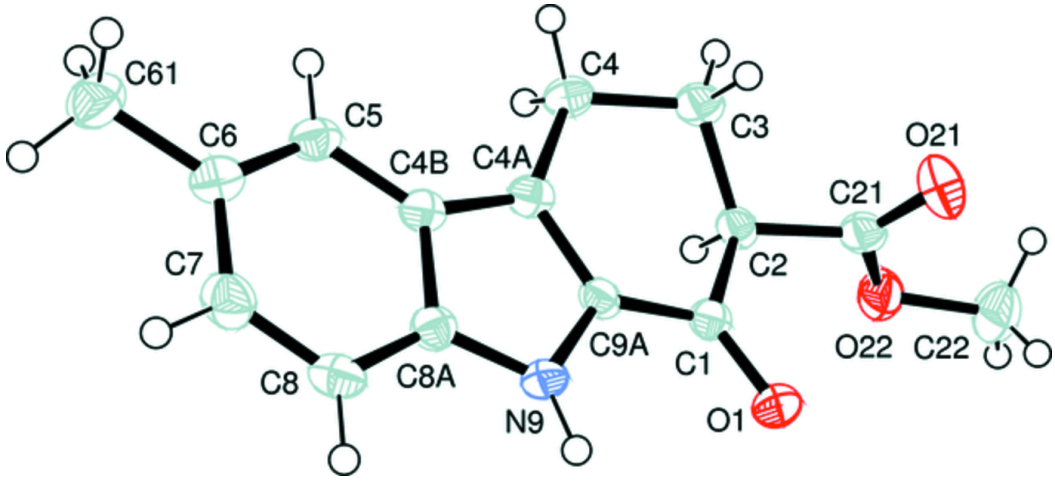

Fig. 2