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Methyl 9-methyl-1-oxo-2,3,4,9-tetrahydro-1*H*-carbazole-2-carboxylate

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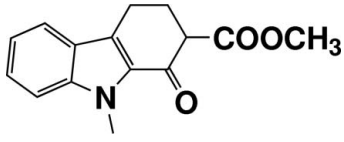
Key indicators: single-crystal X-ray study; T = 203 K; mean σ(C–C) = 0.003 Å; R factor = 0.067; wR factor = 0.177; data-to-parameter ratio = 21.0.

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 0.9 (1)°. The cyclohexene ring is in a half-chair form. The ester group has an equatorial orientation. In the crystal structure, the molecules are stabilized by intermolecular C—H···O hydrogen bonds.

Related literature

For related literature, see: Knolker & Reddy (2002); Gunaseelan *et al.* (2007*a*,*b*). Due to the interesting and important properties of carbazoles, a number of methodologies for the construction of the carbazole ring with other heterocyclic compounds have been reported (Knolker & Reddy, 2002). Gunaseelan *et al.* (2007*a*,*b*) have reported crystal structures of substituted carbazole derivatives, wherein the carbazole units are not planar.



Experimental

Crystal data

$C_{15}H_{15}NO_3$
M_r = 257.28
Monoclinic, $P2_1/a$
a = 9.1267 (3) Å
b = 8.2114 (3) Å
c = 17.2304 (9) Å
β = 91.151 (4)°
V = 1291.04 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 203 (2) K
0.67 × 0.35 × 0.32 mm

Data collection

Oxford Diffraction Gemini diffractometer
Absorption correction: none
13753 measured reflections
3646 independent reflections
2639 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.177
S = 1.14
3646 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9A···O21^{i}	0.97	2.57	3.372 (3)	140
C22—H22B···O1^{ii}	0.97	2.37	3.321 (3)	167

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2007); 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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2018).

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supplementary materials

Acta Cryst. (2007). E**63**, o3093 [**doi:10.1107/S160053680702644X**]

Methyl 9-methyl-1-oxo-2,3,4,9-tetrahydro-1*H*-carbazole-2-carboxylate

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Comment

The molecular structure of (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 0.9 (1)°. The cyclohexene ring is in half-chair form. In the crystal structure, the molecules are stabilized by intermolecular C9—H9A···O21(x + 1, y, z) and C22—H22B···O1(−x + 1/2, y + 1/2, −z) hydrogen bonds (Fig. 2).

Experimental

The mixture of 9-methyl-1,2,3,4-tetrahydrocarbazol-1-one (200 mg, 0.001 mol), dimethyl carbonate (2 ml), sodium hydride in mineral oil (300 mg), catalytic amount of potassium hydride (0.030 mg) [CAUTION: Potassium hydride is highly pyrophoric in dry condition] was refluxed on a water bath for 3 h. After cooling the mixture was carefully neutralized using glacial acetic acid and then poured into crushed ice. It was extracted with ethyl acetate and purified by column chromatography over silica gel using petroleum ether/ethyl acetate (98:2) as eluant to get the pure compound (I) (205 mg, 80%). It was recrystallized using glacial acetic acid.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2 to 1.5 times U_{eq}(C).

Figures

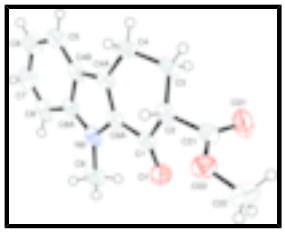

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

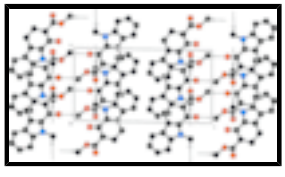

Fig. 2. The molecular packing of (I), viewed down the *b* axis. Dashed lines indicate hydrogen bonds.

Methyl 9-methyl-1-oxo-2,3,4,9-tetrahydro-1*H*-carbazole-2-carboxylate

Crystal data

$C_{15}H_{15}NO_3$	F_{000} = 544
M_r = 257.28	D_x = 1.324 Mg m^{-3}
Monoclinic, $P2_1/a$	Melting point: 361(1) K
Hall symbol: -P 2yab	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 9.1267 (3) Å	Cell parameters from 7041 reflections
b = 8.2114 (3) Å	θ = 4.7–30.7º
c = 17.2304 (9) Å	μ = 0.09 mm^{-1}
β = 91.151 (4)º	T = 203 (2) K
V = 1291.04 (9) Å^3	Plate, colourless
Z = 4	0.67 × 0.35 × 0.32 mm

Data collection

Oxford Diffraction Gemini diffractometer	2639 reflections with $I > 2\sigma(I)$
Radiation source: Enhance (Mo) X-ray Source	R_{int} = 0.038
Monochromator: graphite	θ_{max} = 30.7º
T = 203(2) K	θ_{min} = 4.7º
φ and ω scans	h = −13→12
Absorption correction: none	k = −11→10
13753 measured reflections	l = −23→23
3646 independent reflections	Standard reflections: ?

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.067	H-atom parameters constrained
$wR(F^2)$ = 0.177	$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.8204P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.14	$(\Delta/\sigma)_{max} < 0.001$
3646 reflections	$\Delta\rho_{max}$ = 0.35 e Å^{-3}
174 parameters	$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > 2\text{sigma}(F^2)$ 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	0.41622 (18)	0.4836 (2)	0.11376 (10)	0.0517 (5)
O21	0.06438 (19)	0.5261 (2)	0.15535 (11)	0.0624 (7)
O22	0.17217 (18)	0.7308 (2)	0.09356 (9)	0.0506 (5)
N9	0.66243 (17)	0.4350 (2)	0.22709 (11)	0.0383 (5)
C1	0.4145 (2)	0.5367 (2)	0.17927 (12)	0.0360 (6)
C2	0.2865 (2)	0.6423 (2)	0.20840 (12)	0.0375 (6)
C3	0.2474 (2)	0.5956 (3)	0.29114 (13)	0.0439 (6)
C4	0.3767 (2)	0.6254 (3)	0.34675 (12)	0.0444 (7)
C4A	0.5123 (2)	0.5489 (2)	0.31546 (12)	0.0359 (6)
C4B	0.6424 (2)	0.4969 (2)	0.35477 (12)	0.0380 (6)
C5	0.6906 (3)	0.5009 (3)	0.43246 (14)	0.0485 (7)
C6	0.8261 (3)	0.4360 (3)	0.45130 (16)	0.0572 (8)
C7	0.9150 (3)	0.3685 (3)	0.39403 (17)	0.0557 (9)
C8	0.8729 (2)	0.3644 (3)	0.31774 (16)	0.0486 (8)
C8A	0.7334 (2)	0.4271 (2)	0.29785 (13)	0.0383 (6)
C9	0.7206 (2)	0.3755 (3)	0.15384 (14)	0.0473 (7)
C9A	0.5280 (2)	0.5097 (2)	0.23855 (12)	0.0348 (6)
C21	0.1608 (2)	0.6243 (3)	0.15081 (13)	0.0418 (6)
C22	0.0692 (3)	0.7132 (4)	0.02922 (15)	0.0645 (10)
H2	0.31830	0.75754	0.20847	0.0450*
H3A	0.16314	0.66014	0.30756	0.0526*
H3B	0.21973	0.48036	0.29273	0.0526*
H4A	0.39199	0.74274	0.35328	0.0533*
H4B	0.35560	0.57871	0.39767	0.0533*
H5	0.63187	0.54680	0.47094	0.0583*
H6	0.85943	0.43700	0.50328	0.0686*
H7	1.00655	0.32471	0.40881	0.0669*
H8	0.93460	0.32131	0.27981	0.0584*
H9A	0.82570	0.36047	0.15935	0.0709*
H9B	0.69992	0.45422	0.11309	0.0709*
H9C	0.67470	0.27242	0.14052	0.0709*
H22A	0.08888	0.61257	0.00187	0.0968*
H22B	0.07942	0.80450	−0.00599	0.0968*
H22C	−0.02982	0.71084	0.04863	0.0968*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0440 (9)	0.0605 (10)	0.0508 (9)	−0.0004 (7)	0.0052 (7)	−0.0159 (8)
O21	0.0402 (9)	0.0697 (12)	0.0770 (13)	−0.0125 (8)	−0.0038 (8)	0.0176 (10)
O22	0.0592 (10)	0.0453 (9)	0.0472 (9)	−0.0008 (8)	−0.0009 (7)	0.0056 (7)
N9	0.0304 (8)	0.0309 (8)	0.0540 (11)	0.0005 (6)	0.0114 (7)	−0.0041 (7)
C1	0.0320 (9)	0.0334 (9)	0.0430 (11)	−0.0033 (7)	0.0087 (8)	−0.0001 (8)
C2	0.0336 (9)	0.0316 (9)	0.0475 (11)	0.0045 (7)	0.0060 (8)	0.0026 (8)
C3	0.0311 (9)	0.0505 (12)	0.0504 (12)	0.0118 (9)	0.0100 (8)	0.0041 (10)
C4	0.0383 (10)	0.0569 (13)	0.0383 (11)	0.0125 (9)	0.0099 (8)	0.0017 (9)
C4A	0.0315 (9)	0.0316 (9)	0.0450 (11)	0.0014 (7)	0.0090 (8)	0.0040 (8)
C4B	0.0308 (9)	0.0325 (9)	0.0509 (12)	0.0006 (7)	0.0077 (8)	0.0079 (8)
C5	0.0401 (11)	0.0558 (14)	0.0499 (13)	0.0037 (10)	0.0061 (9)	0.0109 (10)
C6	0.0436 (12)	0.0669 (16)	0.0609 (15)	0.0017 (12)	−0.0019 (11)	0.0159 (12)
C7	0.0348 (11)	0.0501 (14)	0.0822 (19)	0.0085 (10)	−0.0021 (11)	0.0109 (12)
C8	0.0333 (10)	0.0353 (11)	0.0776 (17)	0.0060 (8)	0.0088 (10)	0.0010 (10)
C8A	0.0303 (9)	0.0266 (9)	0.0583 (13)	0.0002 (7)	0.0077 (8)	0.0036 (8)
C9	0.0374 (10)	0.0412 (11)	0.0638 (14)	0.0042 (9)	0.0158 (10)	−0.0125 (10)
C9A	0.0295 (9)	0.0277 (9)	0.0476 (11)	−0.0009 (7)	0.0100 (7)	0.0004 (8)
C21	0.0381 (10)	0.0401 (11)	0.0474 (12)	0.0054 (8)	0.0073 (9)	0.0016 (9)
C22	0.0790 (19)	0.0707 (18)	0.0437 (14)	0.0033 (15)	−0.0046 (12)	0.0015 (12)

Geometric parameters (Å, °)

O1—C1	1.210 (3)	C6—C7	1.404 (4)
O21—C21	1.197 (3)	C7—C8	1.363 (4)
O22—C21	1.324 (3)	C8—C8A	1.409 (3)
O22—C22	1.446 (3)	C2—H2	0.9900
N9—C8A	1.371 (3)	C3—H3A	0.9800
N9—C9	1.463 (3)	C3—H3B	0.9800
N9—C9A	1.389 (2)	C4—H4A	0.9800
C1—C2	1.547 (3)	C4—H4B	0.9800
C1—C9A	1.457 (3)	C5—H5	0.9400
C2—C3	1.526 (3)	C6—H6	0.9400
C2—C21	1.509 (3)	C7—H7	0.9400
C3—C4	1.525 (3)	C8—H8	0.9400
C4—C4A	1.498 (3)	C9—H9A	0.9700
C4A—C4B	1.421 (3)	C9—H9B	0.9700
C4A—C9A	1.374 (3)	C9—H9C	0.9700
C4B—C5	1.401 (3)	C22—H22A	0.9700
C4B—C8A	1.419 (3)	C22—H22B	0.9700
C5—C6	1.379 (4)	C22—H22C	0.9700
O1···O22	3.028 (2)	C9···H8	2.9200
O1···N9	2.974 (2)	C9A···H8^{v}	2.9400
O1···C9	2.984 (3)	C9A···H3B	2.9900
O1···C22^{i}	3.217 (3)	C22···H22A^{ix}	3.0800

O1···C22^{ii}	3.321 (3)	H2···C4A	3.0600
O21···C9^{iii}	3.372 (3)	H2···H4A	2.5700
O22···C1^{iv}	3.391 (2)	H2···N9^{iv}	2.9200
O22···O1	3.028 (2)	H3A···C4A^{iv}	2.7600
O1···H22B^{ii}	2.3700	H3A···C4B^{iv}	2.9400
O1···H9B	2.6000	H3B···O21	2.7600
O1···H22C^{i}	2.8000	H3B···C9A	2.9900
O21···H9A^{iii}	2.5700	H4A···H2	2.5700
O21···H3B	2.7600	H4B···C5^{viii}	3.0400
O21···H9C^{v}	2.6600	H4B···H5^{viii}	2.4900
O21···H22A	2.7500	H5···H4B^{viii}	2.4900
O21···H22C	2.5200	H7···C4B^{x}	3.0700
O22···H9B^{iv}	2.6200	H8···C9	2.9200
O22···H22C^{i}	2.8800	H8···H9A	2.3100
N9···O1	2.974 (2)	H8···C9A^{x}	2.9400
N9···H2^{i}	2.9200	H9A···O21^{vi}	2.5700
C1···O22^{i}	3.391 (2)	H9A···C8	2.7500
C9···O21^{vi}	3.372 (3)	H9A···H8	2.3100
C9···O1	2.984 (3)	H9B···O1	2.6000
C9A···C21^{i}	3.586 (3)	H9B···C1	2.9400
C21···C9A^{iv}	3.586 (3)	H9B···O22^{i}	2.6200
C22···O1^{iv}	3.217 (3)	H9C···O21^{x}	2.6600
C22···O1^{vii}	3.321 (3)	H22A···O21	2.7500
C1···H9B	2.9400	H22A···C22^{ix}	3.0800
C4A···H2	3.0600	H22A···H22A^{ix}	2.4600
C4A···H3A^{i}	2.7600	H22B···O1^{vii}	2.3700
C4B···H3A^{i}	2.9400	H22C···O21	2.5200
C4B···H7^{v}	3.0700	H22C···O1^{iv}	2.8000
C5···H4B^{viii}	3.0400	H22C···O22^{iv}	2.8800
C8···H9A	2.7500		
C21—O22—C22	116.47 (19)	C3—C2—H2	108.00
C8A—N9—C9	125.34 (16)	C21—C2—H2	108.00
C8A—N9—C9A	107.30 (17)	C2—C3—H3A	109.00
C9—N9—C9A	127.36 (17)	C2—C3—H3B	110.00
O1—C1—C2	121.91 (18)	C4—C3—H3A	109.00
O1—C1—C9A	125.21 (17)	C4—C3—H3B	110.00
C2—C1—C9A	112.88 (17)	H3A—C3—H3B	108.00
C1—C2—C3	110.78 (16)	C3—C4—H4A	110.00
C1—C2—C21	107.51 (16)	C3—C4—H4B	110.00
C3—C2—C21	113.63 (16)	C4A—C4—H4A	110.00
C2—C3—C4	110.75 (16)	C4A—C4—H4B	110.00
C3—C4—C4A	109.94 (17)	H4A—C4—H4B	108.00
C4—C4A—C4B	130.02 (18)	C4B—C5—H5	121.00

C4—C4A—C9A	123.25 (17)	C6—C5—H5	121.00
C4B—C4A—C9A	106.68 (16)	C5—C6—H6	119.00
C4A—C4B—C5	133.8 (2)	C7—C6—H6	120.00
C4A—C4B—C8A	106.68 (18)	C6—C7—H7	119.00
C5—C4B—C8A	119.54 (19)	C8—C7—H7	119.00
C4B—C5—C6	118.6 (2)	C7—C8—H8	121.00
C5—C6—C7	121.0 (2)	C8A—C8—H8	121.00
C6—C7—C8	122.1 (2)	N9—C9—H9A	109.00
C7—C8—C8A	117.5 (2)	N9—C9—H9B	109.00
N9—C8A—C4B	108.85 (16)	N9—C9—H9C	109.00
N9—C8A—C8	130.0 (2)	H9A—C9—H9B	109.00
C4B—C8A—C8	121.2 (2)	H9A—C9—H9C	109.00
N9—C9A—C1	125.80 (18)	H9B—C9—H9C	109.00
N9—C9A—C4A	110.49 (17)	O22—C22—H22A	110.00
C1—C9A—C4A	123.64 (17)	O22—C22—H22B	109.00
O21—C21—O22	124.2 (2)	O22—C22—H22C	109.00
O21—C21—C2	125.0 (2)	H22A—C22—H22B	109.00
O22—C21—C2	110.82 (17)	H22A—C22—H22C	109.00
C1—C2—H2	108.00	H22B—C22—H22C	109.00
C22—O22—C21—C2	172.37 (19)	C1—C2—C3—C4	61.9 (2)
C22—O22—C21—O21	−6.8 (3)	C2—C3—C4—C4A	−50.2 (2)
C9—N9—C8A—C8	0.4 (3)	C3—C4—C4A—C9A	20.5 (3)
C9A—N9—C8A—C4B	0.30 (19)	C3—C4—C4A—C4B	−156.62 (19)
C8A—N9—C9A—C1	−177.24 (16)	C4—C4A—C4B—C5	−1.6 (4)
C8A—N9—C9A—C4A	−0.2 (2)	C9A—C4A—C4B—C8A	0.19 (19)
C9A—N9—C8A—C8	−179.3 (2)	C4—C4A—C4B—C8A	177.68 (19)
C9—N9—C8A—C4B	180.00 (17)	C9A—C4A—C4B—C5	−179.1 (2)
C9—N9—C9A—C4A	−179.87 (19)	C4B—C4A—C9A—N9	0.0 (2)
C9—N9—C9A—C1	3.1 (3)	C4B—C4A—C9A—C1	177.13 (16)
O1—C1—C2—C21	14.5 (2)	C4—C4A—C9A—N9	−177.72 (17)
O1—C1—C2—C3	139.23 (19)	C4—C4A—C9A—C1	−0.6 (3)
O1—C1—C9A—N9	7.6 (3)	C4A—C4B—C5—C6	179.1 (2)
C9A—C1—C2—C3	−40.5 (2)	C4A—C4B—C8A—C8	179.35 (18)
C9A—C1—C2—C21	−165.21 (16)	C8A—C4B—C5—C6	−0.1 (3)
C2—C1—C9A—C4A	10.6 (2)	C4A—C4B—C8A—N9	−0.30 (19)
C2—C1—C9A—N9	−172.67 (16)	C5—C4B—C8A—C8	−1.3 (3)
O1—C1—C9A—C4A	−169.09 (18)	C5—C4B—C8A—N9	179.10 (18)
C21—C2—C3—C4	−176.95 (18)	C4B—C5—C6—C7	0.6 (4)
C1—C2—C21—O21	92.4 (2)	C5—C6—C7—C8	0.3 (4)
C3—C2—C21—O21	−30.6 (3)	C6—C7—C8—C8A	−1.7 (4)
C3—C2—C21—O22	150.21 (18)	C7—C8—C8A—C4B	2.1 (3)
C1—C2—C21—O22	−86.8 (2)	C7—C8—C8A—N9	−178.3 (2)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9A···O21^{vi}	0.97	2.57	3.372 (3)	140

C22—H22B···O1^{vii}	0.97	2.37	3.321 (3)	167

Symmetry codes: (vi) $x+1, y, z$; (vii) $-x+1/2, y+1/2, -z$.

Fig. 1

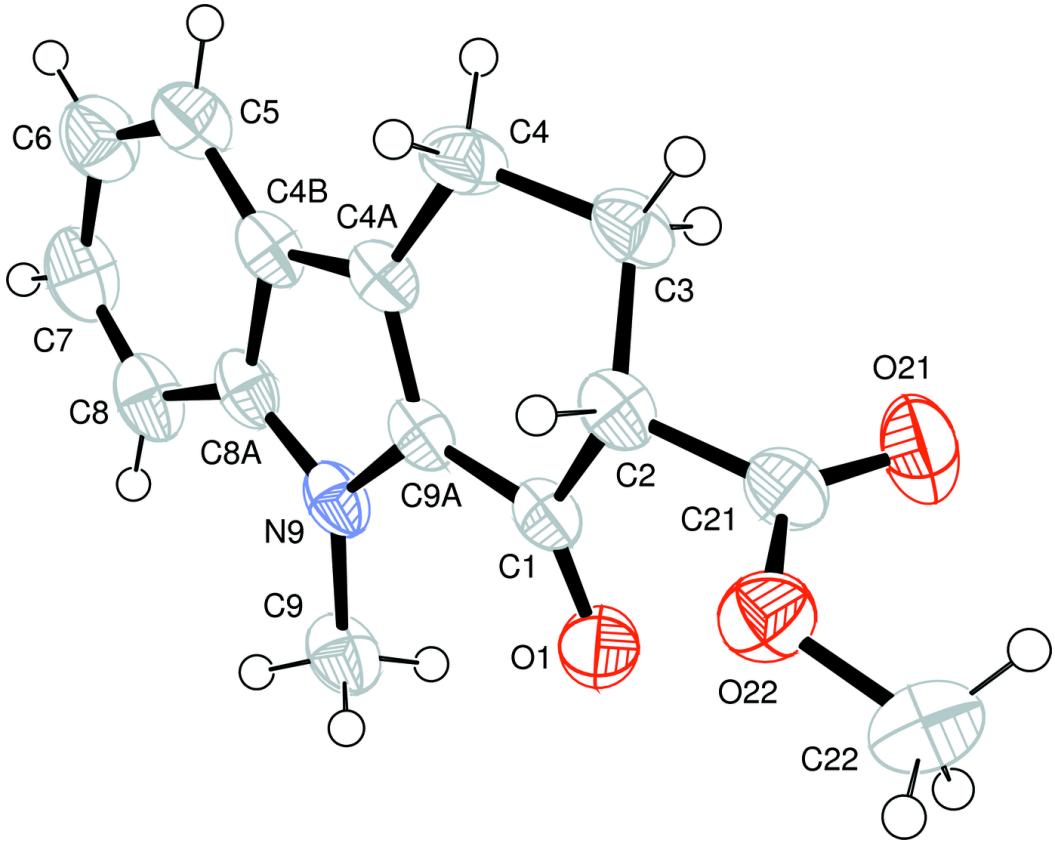

Fig. 2