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

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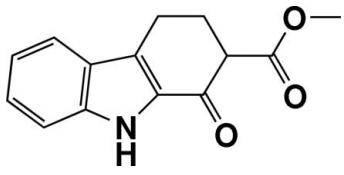
Key indicators: single-crystal X-ray study; T = 203 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.067; wR factor = 0.221; data-to-parameter ratio = 18.0.

In the title molecule, $C_{14}H_{13}NO_3$, the dihedral angle between the benzene ring and the fused pyrrole ring is 1.0 (1)°. The cyclohexene ring adopts a twist conformation. Three C atoms of the cyclohexene ring, with their attached H atoms, and all atoms of the ester group, are disordered over two positions; the site-occupancy factors are *ca* 0.88 and 0.12. The crystal structure is stabilized by intermolecular N—H···O and C—H···O hydrogen bonds.

Related literature

For related literature, see: Gunaseelan *et al.* (2007*a*,*b*); Thiruvalluvar *et al.* (2007).



Experimental

Crystal data

$C_{14}H_{13}NO_3$
M_r = 243.25
Triclinic, $P\overline{1}$
a = 6.9814 (12) Å
b = 8.1303 (13) Å
c = 10.780 (2) Å
α = 88.575 (14)°
β = 76.881 (15)°
γ = 79.367 (14)°
V = 585.59 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 203 (2) K
0.36 × 0.31 × 0.22 mm

Data collection

Oxford Diffraction Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.890, T_{max} = 1.000 (expected range 0.872–0.980)
6572 measured reflections
3176 independent reflections
1673 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.221
S = 1.11
3176 reflections
176 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.80 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 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}	0.87	2.07	2.883 (2)	155
C14A—H14B···O2A^{ii}	0.97	2.39	3.337 (3)	166

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

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

RJB acknowledges the NSF–MRI program for funding to purchase the diffractometer. 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: WN2176).

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  doi:10.1107/S1600536807035660

supplementary materials

Acta Cryst. (2007). E**63**, o3622 [**doi:10.1107/S1600536807035660**]

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

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Comment

The title compound, has been analysed as part of our crystallographic studies on substituted carbazoles (Gunaseelan *et al.* (2007*a*, b); Thiruvalluvar *et al.* (2007)).

The molecular structure of the title compound, with the atomic numbering scheme, is shown in Fig. 1. The dihedral angle between the benzene ring and the fused pyrrole ring is 1.0 (1)°. The cyclohexene ring adopts a twist conformation. The crystal structure is stabilized by intermolecular N9—H9···O1(−*x* + 1,-*y* + 1,-*z*) and C14A—H14B···O2A(−*x* + 1,-*y* + 1,-*z* + 1) hydrogen bonds (Fig. 2).

Experimental

A mixture of methyl 2-(1-oxo-2,3,4,9-tetrahydro-1*H*-carbazol-2-yl) oxoacetate (270 mg, 0.001 mol), glass powder (500 mg) and iron powder (500 mg) in diphenyl ether (15 ml) was heated at 443–473 K for 30 min. Carbon monoxide was 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 to obtain the pure title compound (100 mg, 40%), which was recrystallized from glacial acetic acid.

Refinement

Atoms C2A, C3A, C4A of the cyclohexene ring, with attached hydrogen atoms, and all atoms of the ester group are disordered over two positions; the site occupancy factors refined to 0.879 (3) and 0.121 (3). All H atoms were positioned geometrically and allowed to ride on their parent atoms, with N—H = 0.87 Å, C—H = 0.94–0.99 Å and U_{iso}(H) = xU_{eq}(parent atom), where x = 1.2 or 1.5.

Figures

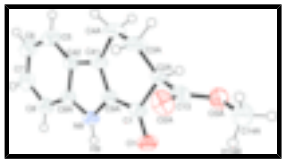

Fig. 1. The molecular structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level. H atoms involved in hydrogen bonding have been labelled. Only the major disorder component is shown.

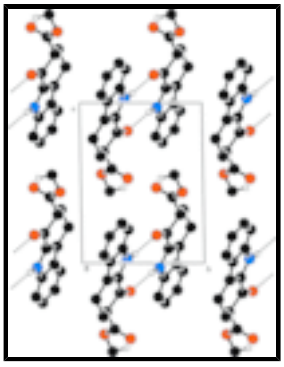

Fig. 2. The molecular packing of the title compound, viewed down the *a* axis. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted. Only the major disorder component is shown.

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

Crystal data

$C_{14}H_{13}NO_3$	$Z = 2$
$M_r = 243.25$	$F_{000} = 256$
Triclinic, $P\overline{1}$	$D_x = 1.380$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 409(1) K
$a = 6.9814$ (12) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 8.1303$ (13) Å	Cell parameters from 2541 reflections
$c = 10.780$ (2) Å	$\theta = 4.8$–$30.6°$
$\alpha = 88.575$ (14)°	$\mu = 0.10$ mm^{-1}
$\beta = 76.881$ (15)°	$T = 203$ (2) K
$\gamma = 79.367$ (14)°	Plate, light-brown
$V = 585.59$ (18) Å^3	0.36 × 0.31 × 0.22 mm

Data collection

Oxford Diffraction Gemini diffractometer	3176 independent reflections
Radiation source: fine-focus sealed tube	1673 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.023$
$T = 203(2)$ K	$\theta_{max} = 30.7°$
φ and ω scans	$\theta_{min} = 4.8°$
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)	$h = -9 \rightarrow 9$
$T_{min} = 0.890$, $T_{max} = 1.000$	$k = -11 \rightarrow 11$
6572 measured reflections	$l = -12 \rightarrow 14$

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

$wR(F^2) = 0.221$	$w = 1/[\sigma^2(F_o^2) + (0.1197P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} < 0.001$
3176 reflections	$\Delta\rho_{max} = 0.80$ e Å^{-3}
176 parameters	$\Delta\rho_{min} = -0.44$ e Å^{-3}
6 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}	Occ. (<1)
C1	0.2838 (3)	0.6953 (3)	0.1859 (2)	0.0373 (5)	
O1	0.4523 (2)	0.6125 (2)	0.17505 (15)	0.0468 (5)	
C5	−0.2646 (3)	0.8206 (3)	−0.0495 (2)	0.0478 (6)	
H5	−0.3758	0.8873	0.0038	0.057*	
C6	−0.2759 (4)	0.7722 (3)	−0.1673 (3)	0.0521 (7)	
H6	−0.3949	0.8067	−0.1952	0.063*	
C7	−0.1117 (4)	0.6713 (3)	−0.2474 (2)	0.0493 (6)	
H7	−0.1228	0.6397	−0.3284	0.059*	
C8	0.0649 (3)	0.6177 (3)	−0.2100 (2)	0.0412 (6)	
H8	0.1742	0.5498	−0.2640	0.049*	
C8A	0.0767 (3)	0.6671 (3)	−0.0902 (2)	0.0348 (5)	
N9	0.2302 (2)	0.6317 (2)	−0.02795 (16)	0.0356 (5)	
H9	0.3471	0.5707	−0.0583	0.043*	
C9A	0.1692 (3)	0.7085 (3)	0.0902 (2)	0.0357 (5)	
C41	−0.0244 (3)	0.7963 (3)	0.1053 (2)	0.0376 (5)	
C42	−0.0870 (3)	0.7707 (3)	−0.0082 (2)	0.0362 (5)	
C13	0.2754 (3)	0.7454 (3)	0.4124 (2)	0.0490 (7)	
C2A	0.1862 (4)	0.8086 (4)	0.3039 (3)	0.0398 (7)	0.879 (3)
H2A	0.2226	0.9198	0.2832	0.048*	0.879 (3)
C3A	−0.0392 (4)	0.8347 (4)	0.3339 (3)	0.0579 (8)	0.879 (3)
H31A	−0.0801	0.7290	0.3642	0.069*	0.879 (3)
H31B	−0.0918	0.9171	0.4037	0.069*	0.879 (3)
C4A	−0.1345 (4)	0.8929 (3)	0.2245 (2)	0.0503 (7)	0.879 (3)
H4A	−0.2742	0.8778	0.2451	0.060*	0.879 (3)
H4B	−0.1330	1.0124	0.2112	0.060*	0.879 (3)

O2A	0.2322 (3)	0.6162 (2)	0.47104 (18)	0.0558 (6)	0.879 (3)
O3A	0.3986 (3)	0.8286 (3)	0.4441 (2)	0.0502 (6)	0.879 (3)
C14A	0.4832 (4)	0.7645 (4)	0.5509 (3)	0.0583 (7)	0.879 (3)
H14A	0.5682	0.8382	0.5692	0.087*	0.879 (3)
H14B	0.5618	0.6533	0.5297	0.087*	0.879 (3)
H14C	0.3760	0.7591	0.6252	0.087*	0.879 (3)
C2B	0.146 (3)	0.742 (3)	0.325 (2)	0.0398 (7)	0.121 (3)
H2B	0.1077	0.6313	0.3477	0.048*	0.121 (3)
C3B	−0.0392 (4)	0.8347 (4)	0.3339 (3)	0.0579 (8)	0.121 (3)
H32A	−0.0423	0.9362	0.3819	0.069*	0.121 (3)
H32B	−0.1319	0.7723	0.3890	0.069*	0.121 (3)
C4B	−0.1345 (4)	0.8929 (3)	0.2245 (2)	0.0503 (7)	0.121 (3)
H41	−0.2742	0.8778	0.2451	0.060*	0.121 (3)
H42	−0.1330	1.0124	0.2112	0.060*	0.121 (3)
O2B	0.347 (2)	0.8761 (13)	0.3797 (16)	0.0558 (6)	0.121 (3)
O3B	0.3399 (16)	0.6820 (11)	0.5095 (8)	0.0502 (6)	0.121 (3)
C14B	0.4832 (4)	0.7645 (4)	0.5509 (3)	0.0583 (7)	0.121 (3)
H14D	0.4959	0.7267	0.6353	0.087*	0.121 (3)
H14E	0.4370	0.8847	0.5535	0.087*	0.121 (3)
H14F	0.6123	0.7370	0.4917	0.087*	0.121 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0329 (11)	0.0413 (12)	0.0319 (12)	−0.0019 (9)	0.0017 (9)	−0.0063 (9)
O1	0.0336 (8)	0.0562 (10)	0.0431 (10)	0.0059 (7)	−0.0037 (7)	−0.0127 (8)
C5	0.0379 (12)	0.0466 (14)	0.0535 (16)	0.0005 (10)	−0.0071 (11)	0.0114 (11)
C6	0.0425 (13)	0.0549 (15)	0.0598 (17)	−0.0063 (12)	−0.0172 (12)	0.0173 (13)
C7	0.0574 (15)	0.0525 (14)	0.0437 (14)	−0.0184 (12)	−0.0173 (11)	0.0063 (11)
C8	0.0435 (12)	0.0370 (12)	0.0412 (13)	−0.0080 (10)	−0.0058 (10)	0.0030 (10)
C8A	0.0357 (11)	0.0313 (11)	0.0358 (12)	−0.0073 (8)	−0.0041 (9)	0.0040 (9)
N9	0.0281 (9)	0.0399 (10)	0.0341 (10)	−0.0007 (7)	−0.0009 (7)	−0.0058 (8)
C9A	0.0332 (11)	0.0326 (11)	0.0366 (12)	−0.0026 (9)	−0.0009 (9)	−0.0033 (9)
C41	0.0324 (11)	0.0320 (11)	0.0404 (12)	0.0035 (8)	0.0009 (9)	−0.0019 (9)
C42	0.0328 (11)	0.0319 (11)	0.0392 (12)	−0.0011 (8)	−0.0030 (9)	0.0060 (9)
C13	0.0426 (13)	0.0516 (15)	0.0446 (14)	−0.0052 (11)	0.0066 (10)	−0.0231 (12)
C2A	0.0448 (15)	0.0333 (15)	0.0356 (14)	−0.0027 (12)	−0.0004 (11)	−0.0086 (12)
C3A	0.0425 (13)	0.0701 (18)	0.0487 (15)	0.0139 (12)	−0.0021 (11)	−0.0228 (13)
C4A	0.0436 (13)	0.0478 (14)	0.0471 (15)	0.0086 (11)	0.0021 (11)	−0.0067 (11)
O2A	0.0706 (14)	0.0513 (12)	0.0476 (12)	−0.0167 (10)	−0.0134 (10)	0.0046 (10)
O3A	0.0525 (12)	0.0491 (11)	0.0515 (13)	−0.0132 (9)	−0.0128 (9)	−0.0048 (10)
C14A	0.0597 (16)	0.0636 (17)	0.0539 (16)	−0.0092 (13)	−0.0189 (13)	−0.0027 (13)
C2B	0.0448 (15)	0.0333 (15)	0.0356 (14)	−0.0027 (12)	−0.0004 (11)	−0.0086 (12)
C3B	0.0425 (13)	0.0701 (18)	0.0487 (15)	0.0139 (12)	−0.0021 (11)	−0.0228 (13)
C4B	0.0436 (13)	0.0478 (14)	0.0471 (15)	0.0086 (11)	0.0021 (11)	−0.0067 (11)
O2B	0.0706 (14)	0.0513 (12)	0.0476 (12)	−0.0167 (10)	−0.0134 (10)	0.0046 (10)
O3B	0.0525 (12)	0.0491 (11)	0.0515 (13)	−0.0132 (9)	−0.0128 (9)	−0.0048 (10)
C14B	0.0597 (16)	0.0636 (17)	0.0539 (16)	−0.0092 (13)	−0.0189 (13)	−0.0027 (13)

Geometric parameters (Å, °)

O1—C1	1.225 (3)	C8—C8A	1.386 (3)
O2A—C13	1.262 (3)	C8A—C42	1.422 (3)
O2B—C13	1.265 (12)	C9A—C41	1.383 (3)
O3A—C13	1.293 (3)	C41—C42	1.422 (3)
O3A—C14A	1.454 (4)	C2A—H2A	0.9900
O3B—C13	1.290 (10)	C2B—H2B	0.9900
O3B—C14B	1.455 (11)	C3A—H31A	0.9800
N9—C8A	1.373 (3)	C3A—H31B	0.9800
N9—C9A	1.374 (3)	C3B—H32A	0.9800
N9—H9	0.8700	C3B—H32B	0.9800
C1—C2B	1.60 (2)	C4A—H4A	0.9800
C1—C2A	1.538 (4)	C4A—H4B	0.9800
C1—C9A	1.433 (3)	C4B—H41	0.9800
C2A—C13	1.485 (4)	C4B—H42	0.9800
C2A—C3A	1.508 (4)	C5—H5	0.9400
C2B—C3B	1.36 (2)	C6—H6	0.9400
C2B—C13	1.45 (2)	C7—H7	0.9400
C3A—C4A	1.506 (4)	C8—H8	0.9400
C3B—C4B	1.506 (4)	C14A—H14B	0.9700
C4A—C41	1.497 (3)	C14A—H14C	0.9700
C4B—C41	1.497 (3)	C14A—H14A	0.9700
C5—C42	1.398 (3)	C14B—H14D	0.9700
C5—C6	1.362 (4)	C14B—H14E	0.9700
C6—C7	1.404 (4)	C14B—H14F	0.9700
C7—C8	1.376 (4)		
O1···O2A	3.210 (3)	C9A···H31A	3.0600
O1···N9	2.941 (2)	C14A···H6^{xiii}	3.0900
O1···C5^{i}	3.389 (3)	C14B···H6^{xiii}	3.0900
O1···O2B	2.986 (14)	C14B···H32B^{i}	2.8600
O1···N9^{ii}	2.883 (2)	C42···H4B^{xi}	3.0900
O2A···C8^{iii}	3.371 (3)	C42···H42^{xi}	3.0900
O2A···C14A^{iv}	3.337 (3)	H2A···C6^{xi}	2.8200
O2A···O1	3.210 (3)	H2B···C7^{vii}	2.7000
O2A···C7^{iii}	3.397 (4)	H2B···C3A	1.8000
O2B···O1	2.986 (14)	H2B···C4A	2.9500
O3B···C8^{iii}	3.280 (10)	H2B···H7^{vii}	2.2000
O1···H9^{ii}	2.0700	H4A···O3A^{viii}	2.8400
O1···H9	2.8200	H4B···C8A^{xi}	2.9400
O2A···H8^{iii}	2.8400	H4B···C42^{xi}	3.0900
O2A···H31A	2.7000	H5···C5^{x}	3.0800
O2A···H14B	2.5900	H5···H5^{x}	2.2900
O2A···H14C	2.5300	H6···C14B^{xii}	3.0900
O2A···H14B^{iv}	2.3900	H6···H14D^{xii}	2.2800

O2A···H7^{iii}	2.8800	H6···C14A^{xii}	3.0900
O2B···H14E^{v}	2.8700	H7···O2A^{ix}	2.8800
O2B···H14F	2.5200	H7···H2B^{vii}	2.2000
O2B···H14E	2.1100	H8···O3B^{ix}	2.7400
O2B···H32A	2.6700	H8···O2A^{ix}	2.8400
O2B···H41^{i}	2.7200	H8···H14C^{ix}	2.5200
O3A···H31B^{vi}	2.9100	H9···O1	2.8200
O3A···H14A^{v}	2.7600	H9···O1^{ii}	2.0700
O3A···H4A^{i}	2.8400	H14A···O3A^{v}	2.7600
O3B···H8^{iii}	2.7400	H14B···O2A	2.5900
N9···O1^{ii}	2.883 (2)	H14B···O2A^{iv}	2.3900
N9···O1	2.941 (2)	H14C···O2A	2.5300
C1···C7^{vii}	3.410 (4)	H14C···C8^{iii}	2.8700
C2B···C7^{vii}	3.55 (2)	H14C···H8^{iii}	2.5200
C2B···C4A	2.55 (2)	H14D···C6^{xiii}	3.0000
C5···O1^{viii}	3.389 (3)	H14D···H6^{xiii}	2.2800
C7···C1^{vii}	3.410 (4)	H14E···O2B	2.1100
C7···C9A^{vii}	3.529 (4)	H14E···O2B^{v}	2.8700
C7···O2A^{ix}	3.397 (4)	H14E···H14E^{v}	2.3800
C7···C2B^{vii}	3.55 (2)	H14F···H32B^{i}	1.9400
C8···C9A^{vii}	3.442 (3)	H14F···C3B^{i}	2.8700
C8···O2A^{ix}	3.371 (3)	H14F···O2B	2.5200
C8···C41^{vii}	3.564 (3)	H31A···O2A	2.7000
C8···O3B^{ix}	3.280 (10)	H31A···C9A	3.0600
C8A···C8A^{vii}	3.490 (3)	H31B···O3A^{vi}	2.9100
C9A···C8^{vii}	3.442 (3)	H32A···O2B	2.6700
C9A···C7^{vii}	3.529 (4)	H32A···C4A	2.0100
C14A···O2A^{iv}	3.337 (3)	H32A···H4A	2.5300
C41···C8^{vii}	3.564 (3)	H32A···H4B	2.1200
C2B···H4B	3.0800	H32B···C4A	2.0100
C3B···H4B	2.0500	H32B···C14B^{viii}	2.8600
C3B···H4A	2.0500	H32B···H14F^{viii}	1.9400
C3B···H14F^{viii}	2.8700	H32B···H4A	2.1100
C5···H5^{x}	3.0800	H41···O2B^{viii}	2.7200
C6···H2A^{xi}	2.8200	H41···C3A	2.0500
C6···H14D^{xii}	3.0000	H41···H4B	1.5900
C7···H2B^{vii}	2.7000	H42···C3A	2.0500
C8···H14C^{ix}	2.8700	H42···H4A	1.5900
C8A···H4B^{xi}	2.9400	H42···C8A^{xi}	2.9400
C8A···H42^{xi}	2.9400	H42···C42^{xi}	3.0900
C13—O3A—C14A	116.0 (2)	C3A—C2A—H2A	107.00

C13—O3B—C14B	116.2 (7)	C13—C2A—H2A	107.00
C8A—N9—C9A	108.92 (17)	C13—C2B—H2B	99.00
C9A—N9—H9	126.00	C3B—C2B—H2B	99.00
C8A—N9—H9	126.00	C1—C2B—H2B	99.00
O1—C1—C2A	120.4 (2)	C2A—C3A—H31A	108.00
O1—C1—C9A	125.0 (2)	C2A—C3A—H31B	108.00
C2A—C1—C9A	114.4 (2)	H31A—C3A—H31B	107.00
O1—C1—C2B	119.4 (8)	C4A—C3A—H31A	108.00
C2B—C1—C9A	112.2 (8)	C4A—C3A—H31B	108.00
C1—C2A—C13	109.7 (2)	C2B—C3B—H32A	106.00
C3A—C2A—C13	113.9 (2)	C2B—C3B—H32B	106.00
C1—C2A—C3A	112.7 (2)	C4B—C3B—H32A	106.00
C3B—C2B—C13	126.8 (16)	C4B—C3B—H32B	106.00
C1—C2B—C3B	118.0 (14)	H32A—C3B—H32B	106.00
C1—C2B—C13	108.2 (14)	C3A—C4A—H4A	110.00
C2A—C3A—C4A	115.4 (2)	C41—C4A—H4A	110.00
C2B—C3B—C4B	126.3 (9)	C41—C4A—H4B	109.00
C3A—C4A—C41	110.6 (2)	C3A—C4A—H4B	110.00
C3B—C4B—C41	110.6 (2)	H4A—C4A—H4B	108.00
C6—C5—C42	119.7 (2)	C41—C4B—H42	109.00
C5—C6—C7	120.7 (3)	C3B—C4B—H41	110.00
C6—C7—C8	121.5 (3)	C3B—C4B—H42	110.00
C7—C8—C8A	117.8 (2)	H41—C4B—H42	108.00
N9—C8A—C8	130.6 (2)	C41—C4B—H41	110.00
N9—C8A—C42	107.85 (18)	C6—C5—H5	120.00
C8—C8A—C42	121.6 (2)	C42—C5—H5	120.00
N9—C9A—C1	125.8 (2)	C7—C6—H6	120.00
N9—C9A—C41	109.53 (18)	C5—C6—H6	120.00
C1—C9A—C41	124.7 (2)	C8—C7—H7	119.00
O2A—C13—O3A	121.7 (2)	C6—C7—H7	119.00
O2A—C13—C2A	121.2 (2)	C8A—C8—H8	121.00
O3A—C13—C2A	117.2 (2)	C7—C8—H8	121.00
O2B—C13—O3B	109.2 (9)	H14B—C14A—H14C	109.00
O2B—C13—C2B	102.2 (12)	H14A—C14A—H14C	110.00
O3B—C13—C2B	148.4 (10)	O3A—C14A—H14A	109.00
C4A—C41—C42	130.4 (2)	O3A—C14A—H14B	109.00
C9A—C41—C42	107.00 (19)	O3A—C14A—H14C	109.00
C4B—C41—C9A	122.59 (19)	H14A—C14A—H14B	109.00
C4B—C41—C42	130.4 (2)	O3B—C14B—H14D	109.00
C4A—C41—C9A	122.59 (19)	O3B—C14B—H14E	109.00
C5—C42—C41	134.6 (2)	O3B—C14B—H14F	109.00
C8A—C42—C41	106.70 (19)	H14D—C14B—H14E	110.00
C5—C42—C8A	118.7 (2)	H14D—C14B—H14F	109.00
C1—C2A—H2A	107.00	H14E—C14B—H14F	109.00
C14A—O3A—C13—C2A	179.7 (2)	C3A—C4A—C41—C9A	−19.1 (3)
C14A—O3A—C13—O2A	−0.1 (3)	C3A—C4A—C41—C42	159.3 (3)
C8A—N9—C9A—C1	177.0 (2)	C6—C5—C42—C41	−179.6 (3)
C8A—N9—C9A—C41	−0.7 (3)	C6—C5—C42—C8A	−1.3 (4)
C9A—N9—C8A—C8	−179.4 (2)	C42—C5—C6—C7	0.6 (4)

C9A—N9—C8A—C42	0.3 (2)	C5—C6—C7—C8	0.2 (4)
O1—C1—C9A—N9	−0.4 (4)	C6—C7—C8—C8A	−0.3 (4)
C2A—C1—C9A—C41	−8.4 (3)	C7—C8—C8A—C42	−0.4 (4)
O1—C1—C2A—C13	−24.7 (3)	C7—C8—C8A—N9	179.1 (2)
C9A—C1—C2A—C3A	32.3 (3)	N9—C8A—C42—C5	−178.5 (2)
C9A—C1—C2A—C13	160.4 (2)	N9—C8A—C42—C41	0.3 (3)
O1—C1—C9A—C41	177.0 (2)	C8—C8A—C42—C41	179.9 (2)
C2A—C1—C9A—N9	174.2 (2)	C8—C8A—C42—C5	1.2 (4)
O1—C1—C2A—C3A	−152.8 (2)	N9—C9A—C41—C4A	179.6 (2)
C1—C2A—C13—O3A	105.7 (3)	C1—C9A—C41—C42	−176.9 (2)
C3A—C2A—C13—O2A	53.0 (4)	N9—C9A—C41—C42	0.9 (3)
C3A—C2A—C13—O3A	−126.9 (3)	C1—C9A—C41—C4A	1.9 (4)
C13—C2A—C3A—C4A	−177.9 (2)	C4A—C41—C42—C5	−0.9 (5)
C1—C2A—C13—O2A	−74.4 (3)	C4A—C41—C42—C8A	−179.3 (2)
C1—C2A—C3A—C4A	−52.1 (3)	C9A—C41—C42—C5	177.8 (3)
C2A—C3A—C4A—C41	44.1 (3)	C9A—C41—C42—C8A	−0.7 (3)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N9—H9···O1^{ii}	0.87	2.07	2.883 (2)	155
C14A—H14B···O2A^{iv}	0.97	2.39	3.337 (3)	166

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

Fig. 1

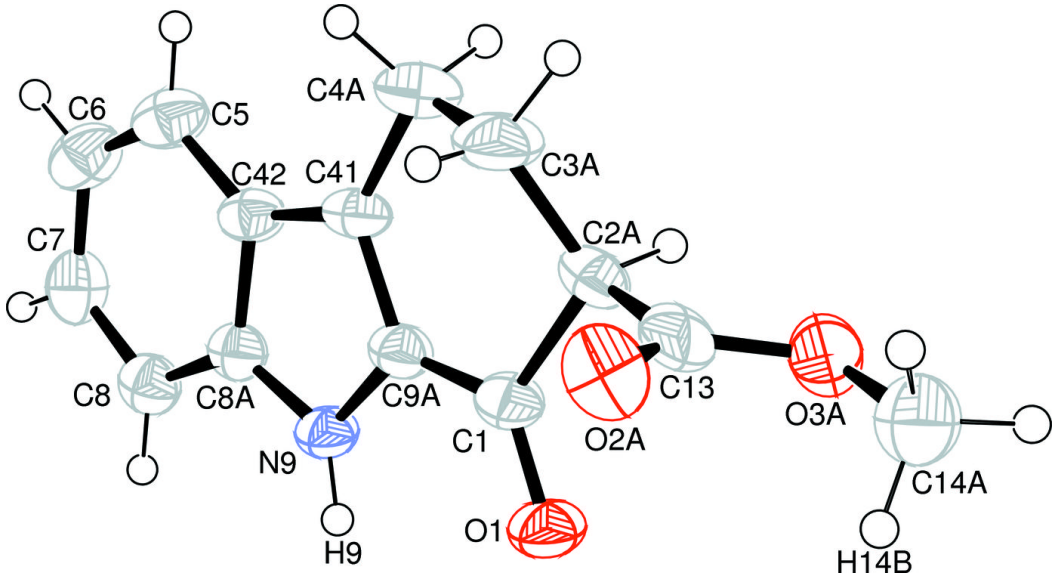
C6
C5
C4A
C42
C41
C3A
C7
C2A
C8
C8A
C9A
C1
C13
O3A
O2A
N9
C14A
O1
H9
H14B

Fig. 2