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Methyl 2,4,5,10-tetrahydropyrazolo-[3,4-*a*]carbazole-3-carboxylate acetic acid solvate at 160 K

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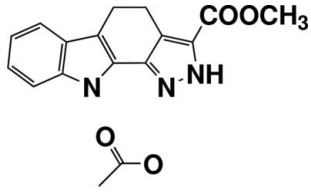
Key indicators: single-crystal X-ray study; T = 160 K; mean σ(C–C) = 0.002 Å; R factor = 0.054; wR factor = 0.152; data-to-parameter ratio = 19.5.

The heterofused carbazole unit of the title compound, $C_{15}H_{13}N_3O_2$, is not planar. The planar pyrazole ring forms dihedral angles of 2.52 (8)°, 3.18 (7)° and 2.22 (6)° with the pyrrole ring, the benzene ring and the attached carboxylate group respectively. The cyclohexene ring adopts a half-chair conformation. In the crystal structure, the molecules are stabilized by intermolecular N—H···O and O—H···N hydrogen bonds.

Related literature

For related literature, see: Balamurali & Rajendra Prasad (2001); Bhattacharayya & Chakraborty (1987); Chakraborty (1993); Chakraborty & Roy (1991); Danish & Rajendra Prasad (2004); Ebenezer & Rajendra Prasad (2006*a*,*b*); Haider (2002); Hewlins *et al.* (1984); Hirata *et al.* (1999); Joule (1984); Kapil (1971); Pinder (1990); Wang *et al.* (2005).



Experimental

Crystal data

$C_{15}H_{13}N_3O_2\cdot C_2H_4O_2$
M_r = 327.34
Triclinic, $P\overline{1}$
a = 7.1379 (2) Å
b = 11.0450 (4) Å
c = 11.5179 (4) Å
α = 117.252 (1)°
β = 100.046 (2)°
γ = 95.486 (2)°
V = 778.75 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 160 (1) K
0.3 × 0.2 × 0.13 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
24031 measured reflections
4507 independent reflections
3773 reflections with $I > 2\sigma(I)$
R_{int} = 0.054

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.152
S = 1.05
4507 reflections
231 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O31^{i}	0.90 (2)	1.95 (2)	2.8181 (14)	163 (2)
O3—H3···N1^{i}	0.97 (3)	1.76 (3)	2.7183 (16)	172 (3)
N10—H10···O2^{i}	0.93 (2)	1.92 (2)	2.8480 (17)	175 (2)

Symmetry code: (i) $-x+1, -y+2, -z+1$.

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: *SIR97* (Altomare *et al.*, 1999); 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 Zürich. His help is gratefully acknowledged by AT, who also 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: BQ2014).

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supplementary materials

Acta Cryst. (2007). E**63**, o2729-o2730 [**doi:10.1107/S1600536807020491**]

Methyl 2,4,5,10-tetrahydropyrazolo[3,4-*a*]carbazole-3-carboxylate acetic acid solvate at 160 K

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Comment

The emerging importance towards the various strategies applied to prepare carbazoles and its derivatives were due to their diverse pharmacological properties (Bhattacharayya *et al.*, 1987; Chakraborty *et al.*, 1991; Chakraborty, 1993; Hewlins *et al.*, 1984). Development of new methods for the synthesis of functionalized carbazoles in particular, is attracting organic chemists due to the discovery of many carbazole alkaloids with varied pharmacological properties (Pinder, 1990; Danish, & Rajendra Prasad, 2004; Ebenezer Martin, & Rajendra Prasad, 2006a; Balamurali, & Rajendra Prasad, 2001; Joule, 1984; Kapil, 1971). Identification of promising antineoplastic activity of ellipticine, tetracyclic compounds of the pyridocarbazole type, have stimulated considerable interest in the field of fused systems (Haider, 2002). In addition, pyridocarbazoles were reported to elicit anti-HIV properties (Hirata *et al.*, 1999; Wang *et al.*, 2005).

At this context, we planned to utilize an intermediate, methyl 2-(1-oxo-2,3,4,9-tetrahydro-1*H*-carbazol-2-yl)-2-oxo-acetate (1) to construct newer fused carbazole. The reaction of (1) with hydrazine hydrate yielded pyrazolocarbazole, either methyl 2,4,5,10-tetrahydropyrazolo[3,4-a]carbazole-3-carboxylate (2) or methyl 1,4,5,10-tetrahydropyrazolo[3,4-a]carbazole-3-carboxylate (3) (Ebenezer Martin & Rajendra Prasad, 2006 b). We present here the X-ray crystal and molecular structure of (2).

The molecular structure of (I), with atomic numbering scheme, is shown in Fig. 1. The pyrazolocarbazole unit is not planar. The carboxylate group at position 3 has a 2.22 (6)° tilt with that of the pyrazole ring. The cyclohexene ring adopts a half-chair conformation. Molecules are linked by intermolecular N—H···O and O—H···N hydrogen bonds. The N2—H2···O31 forms an infinite chain. The O3—H3···N1 and N10—H10···O2 hydrogen bonds between the heterofused carbazole units and the acetic acid solvent molecules form a 9-membered ring closure networks (Fig. 2).

Experimental

The methyl 2-(1-oxo-2,3,4,9-tetrahydro-1*H*-carbazol-2-yl)-2-oxoacetate (243 mg, 0.001 mol) in glacial acetic acid (15 ml) was added hydrazine hydrate (0.1 ml, 0.002 mol) and refluxed on oil bath for 1 h. The reaction was monitored by TLC. After the completion of the reaction it was poured into crushed ice. The precipitate was filtered, washed with water and dried. It was purified by column chromatography over silica gel using petroleum ether: ethyl acetate (85:15) as eluant. The product was characterized as (I). The yield of the isolated product was 181 g (68%).

Refinement

H atoms bonded to N2,N10 and O3 were located in a difference 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_{iso}(H) = 1.2–1.5 times U_{eq}(C).

Figures

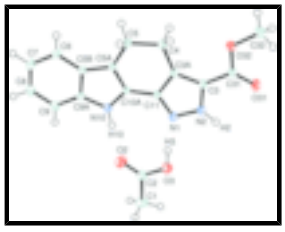

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level. H atoms involved in hydrogen bonding are labelled.

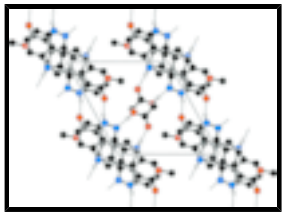

Fig. 2. The packing of (I), viewed down the a axis. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

Methyl 2,4,5,10-tetrahydropyrazolo[3,4-a]carbazole-3-carboxylate acetic acid solvate

Crystal data

$C_{15}H_{13}N_3O_2{\cdot}C_2H_4O_2$	$Z = 2$
$M_r = 327.34$	$F_{000} = 344$
Triclinic, $P\overline{1}$	$D_x = 1.396$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 512(1) K
$a = 7.1379$ (2) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 11.0450$ (4) Å	Cell parameters from 4376 reflections
$c = 11.5179$ (4) Å	$\theta = 2.0$–30.0º
$\alpha = 117.252$ (1)º	$\mu = 0.10$ mm^{-1}
$\beta = 100.046$ (2)º	$T = 160$ (1) K
$\gamma = 95.486$ (2)º	Block, colourless
$V = 778.75$ (5) Å^3	0.3 × 0.2 × 0.13 mm

Data collection

Nonius KappaCCD area-detector diffractometer	4507 independent reflections
Radiation source: Nonius FR590 sealed tube generator	3773 reflections with $I > 2\sigma(I)$
Monochromator: horizontally mounted graphite crystal	$R_{int} = 0.054$
Detector resolution: 9 pixels mm^{-1}	$\theta_{max} = 30.0$º
$T = 160(1)$ K	$\theta_{min} = 2.1$º
φ and ω scans with κ offsets	$h = -10\rightarrow10$
Absorption correction: none	$k = -15\rightarrow15$
24031 measured reflections	$l = -16\rightarrow15$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0862P)^2 + 0.2457P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.055$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.152$	$\Delta\rho_{max} = 0.55$ e Å^{-3}
$S = 1.05$	$\Delta\rho_{min} = -0.24$ e Å^{-3}
4507 reflections	Extinction correction: none
231 parameters	
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.): 0.939 (2) Frames collected: 486 Seconds exposure per frame: 127 Degrees rotation per frame: 1.7 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 $F^2 > 2$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}
O31	0.34994 (16)	0.82581 (10)	0.39653 (10)	0.0314 (3)
O32	0.20500 (14)	0.68331 (9)	0.17749 (9)	0.0264 (3)
N1	0.43529 (15)	1.12962 (10)	0.28289 (10)	0.0206 (3)
N2	0.42196 (16)	1.04370 (11)	0.33625 (10)	0.0217 (3)
N10	0.36245 (16)	1.21943 (11)	0.06189 (11)	0.0219 (3)
C3	0.32604 (17)	0.91281 (12)	0.24179 (12)	0.0196 (3)
C3A	0.27148 (16)	0.91219 (12)	0.12022 (11)	0.0173 (3)
C4	0.15818 (18)	0.80167 (12)	−0.01701 (12)	0.0209 (3)
C5	0.15172 (19)	0.84277 (13)	−0.12920 (12)	0.0228 (3)
C5A	0.21838 (17)	0.99373 (12)	−0.08205 (11)	0.0183 (3)
C5B	0.21568 (17)	1.06733 (13)	−0.15717 (12)	0.0197 (3)
C6	0.1484 (2)	1.02927 (15)	−0.29372 (13)	0.0264 (4)
C7	0.1741 (2)	1.12956 (16)	−0.33302 (15)	0.0323 (4)

C8	0.2638 (2)	1.26784 (16)	−0.23907 (15)	0.0325 (4)
C9	0.3301 (2)	1.30847 (14)	−0.10401 (14)	0.0280 (4)
C9A	0.30722 (18)	1.20748 (13)	−0.06413 (13)	0.0214 (3)
C10A	0.31031 (17)	1.08946 (12)	0.04899 (11)	0.0179 (3)
C11	0.34203 (17)	1.04983 (12)	0.15214 (11)	0.0179 (3)
C31	0.29712 (18)	0.80540 (13)	0.28174 (12)	0.0219 (3)
C32	0.1711 (3)	0.57044 (15)	0.20758 (16)	0.0375 (4)
O2	0.39202 (19)	0.54902 (11)	0.70226 (11)	0.0403 (3)
O3	0.33632 (16)	0.61547 (11)	0.54596 (10)	0.0326 (3)
C1	0.1918 (2)	0.38672 (15)	0.48456 (15)	0.0355 (4)
C2	0.31737 (19)	0.52438 (13)	0.58875 (13)	0.0250 (3)
H2	0.482 (3)	1.070 (2)	0.422 (2)	0.036 (5)*
H4A	0.02342	0.77694	−0.01285	0.0251*
H4B	0.21548	0.71766	−0.04202	0.0251*
H5A	0.23310	0.78999	−0.18831	0.0274*
H5B	0.01653	0.81341	−0.18472	0.0274*
H6	0.08625	0.93623	−0.35802	0.0317*
H7	0.13016	1.10437	−0.42534	0.0387*
H8	0.27922	1.33456	−0.26888	0.0389*
H9	0.38939	1.40228	−0.04033	0.0336*
H10	0.436 (3)	1.298 (2)	0.139 (2)	0.043 (5)*
H32A	0.29511	0.55974	0.25030	0.0561*
H32B	0.11044	0.48403	0.12389	0.0561*
H32C	0.08493	0.59121	0.26892	0.0561*
H1A	0.06179	0.38137	0.50082	0.0532*
H1B	0.18255	0.37707	0.39471	0.0532*
H1C	0.24908	0.31175	0.49005	0.0532*
H3	0.412 (4)	0.706 (3)	0.613 (3)	0.077 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O31	0.0460 (6)	0.0263 (5)	0.0190 (4)	−0.0040 (4)	−0.0014 (4)	0.0140 (4)
O32	0.0369 (5)	0.0179 (4)	0.0204 (4)	−0.0042 (4)	−0.0002 (4)	0.0102 (3)
N1	0.0278 (5)	0.0163 (5)	0.0155 (5)	0.0004 (4)	0.0011 (4)	0.0082 (4)
N2	0.0305 (5)	0.0175 (5)	0.0143 (5)	−0.0014 (4)	−0.0002 (4)	0.0085 (4)
N10	0.0267 (5)	0.0175 (5)	0.0181 (5)	−0.0035 (4)	−0.0014 (4)	0.0096 (4)
C3	0.0236 (6)	0.0172 (5)	0.0165 (5)	0.0003 (4)	0.0022 (4)	0.0086 (4)
C3A	0.0189 (5)	0.0154 (5)	0.0156 (5)	0.0003 (4)	0.0027 (4)	0.0069 (4)
C4	0.0248 (6)	0.0170 (5)	0.0154 (5)	−0.0031 (4)	−0.0002 (4)	0.0065 (4)
C5	0.0310 (6)	0.0174 (5)	0.0142 (5)	−0.0011 (4)	−0.0006 (4)	0.0061 (4)
C5A	0.0195 (5)	0.0174 (5)	0.0162 (5)	0.0001 (4)	0.0012 (4)	0.0084 (4)
C5B	0.0197 (5)	0.0216 (5)	0.0174 (5)	0.0004 (4)	0.0009 (4)	0.0112 (4)
C6	0.0291 (6)	0.0290 (7)	0.0185 (6)	−0.0010 (5)	−0.0013 (5)	0.0132 (5)
C7	0.0363 (7)	0.0390 (8)	0.0237 (6)	−0.0004 (6)	−0.0014 (5)	0.0214 (6)
C8	0.0353 (7)	0.0361 (8)	0.0321 (7)	−0.0014 (6)	0.0006 (6)	0.0258 (6)
C9	0.0315 (7)	0.0241 (6)	0.0289 (7)	−0.0033 (5)	0.0002 (5)	0.0174 (5)
C9A	0.0216 (6)	0.0222 (6)	0.0208 (6)	−0.0003 (4)	0.0010 (4)	0.0131 (5)

C10A	0.0202 (5)	0.0164 (5)	0.0152 (5)	−0.0009 (4)	0.0002 (4)	0.0083 (4)
C11	0.0204 (5)	0.0163 (5)	0.0155 (5)	0.0011 (4)	0.0021 (4)	0.0078 (4)
C31	0.0266 (6)	0.0192 (5)	0.0177 (5)	−0.0003 (4)	0.0016 (4)	0.0095 (5)
C32	0.0542 (9)	0.0213 (6)	0.0342 (8)	−0.0051 (6)	0.0025 (7)	0.0165 (6)
O2	0.0606 (7)	0.0225 (5)	0.0254 (5)	−0.0078 (5)	−0.0083 (5)	0.0109 (4)
O3	0.0442 (6)	0.0228 (5)	0.0222 (5)	−0.0078 (4)	−0.0027 (4)	0.0103 (4)
C1	0.0420 (8)	0.0213 (6)	0.0267 (7)	−0.0074 (5)	−0.0018 (6)	0.0043 (5)
C2	0.0290 (6)	0.0184 (5)	0.0209 (6)	0.0006 (4)	0.0028 (5)	0.0060 (5)

Geometric parameters (Å, °)

O31—C31	1.2157 (16)	C5B—C9A	1.419 (2)
O32—C31	1.3284 (17)	C6—C7	1.384 (3)
O32—C32	1.449 (2)	C7—C8	1.404 (2)
O2—C2	1.2127 (17)	C8—C9	1.382 (2)
O3—C2	1.313 (2)	C9—C9A	1.394 (2)
O3—H3	0.97 (3)	C10A—C11	1.4338 (18)
N1—C11	1.3455 (15)	C4—H4B	0.9900
N1—N2	1.3508 (18)	C4—H4A	0.9900
N2—C3	1.3612 (18)	C5—H5B	0.9900
N10—C9A	1.3765 (18)	C5—H5A	0.9900
N10—C10A	1.380 (2)	C6—H6	0.9500
N2—H2	0.90 (2)	C7—H7	0.9500
N10—H10	0.93 (2)	C8—H8	0.9500
C3—C31	1.466 (2)	C9—H9	0.9500
C3—C3A	1.3831 (17)	C32—H32C	0.9800
C3A—C11	1.406 (2)	C32—H32B	0.9800
C3A—C4	1.4969 (17)	C32—H32A	0.9800
C4—C5	1.5487 (19)	C1—C2	1.500 (2)
C5—C5A	1.493 (2)	C1—H1A	0.9800
C5A—C10A	1.3736 (16)	C1—H1B	0.9800
C5A—C5B	1.432 (2)	C1—H1C	0.9800
C5B—C6	1.4048 (18)		
O2···N10^{i}	2.8480 (17)	C11···C5A^{vi}	3.3978 (17)
O3···C2^{ii}	3.2828 (18)	C31···C7^{vii}	3.598 (2)
O3···N1^{i}	2.7183 (16)	C31···C8^{vi}	3.373 (2)
O31···N2^{i}	2.8181 (14)	C2···H5A^{iv}	3.0900
O31···N2	2.8207 (18)	C2···H10^{i}	2.93 (2)
O32···C4	3.0568 (17)	C5A···H4A^{vii}	3.0900
O2···H8^{iii}	2.6100	C5B···H4A^{vii}	2.8900
O2···H5A^{iv}	2.8200	C6···H7^{viii}	3.0700
O2···H32A^{ii}	2.7200	C7···H7^{viii}	3.0600
O2···H10^{i}	1.92 (2)	C9A···H4A^{vii}	2.6600
O3···H1A^{v}	2.8100	C10A···H5B^{vii}	3.0400
O31···H32A	2.5800	C10A···H4A^{vii}	2.9900
O31···H2	2.64 (2)	C11···H5B^{vii}	3.0900

O31···H2^{i}	1.95 (2)	C11···H3^{i}	2.90 (3)
O31···H32C	2.6600	C31···H2^{i}	3.07 (2)
O32···H4B	2.7400	H1A···O3^{v}	2.8100
N1···O3^{i}	2.7183 (16)	H1B···H5B^{ix}	2.4200
N1···N10	3.1117 (17)	H2···C31^{i}	3.07 (2)
N2···O31	2.8207 (18)	H2···O31	2.64 (2)
N2···O31^{i}	2.8181 (14)	H2···O31^{i}	1.95 (2)
N2···C6^{vi}	3.3060 (19)	H3···N2^{i}	2.65 (4)
N10···N1	3.1117 (17)	H3···N1^{i}	1.76 (3)
N10···O2^{i}	2.8480 (17)	H3···C11^{i}	2.90 (3)
N1···H3^{i}	1.76 (3)	H4A···N10^{vii}	2.7200
N2···H3^{i}	2.65 (4)	H4A···C5A^{vii}	3.0900
N10···H4A^{vii}	2.7200	H4A···C9A^{vii}	2.6600
C2···O3^{ii}	3.2828 (18)	H4A···C10A^{vii}	2.9900
C2···C2^{ii}	3.5195 (19)	H4A···H32B^{ix}	2.5500
C3···C5B^{vi}	3.5874 (18)	H4A···C5B^{vii}	2.8900
C3···C9A^{vi}	3.5846 (18)	H4B···O32	2.7400
C3A···C9A^{vi}	3.4443 (18)	H5A···O2^{x}	2.8200
C3A···C5B^{vi}	3.5836 (17)	H5A···C2^{x}	3.0900
C3A···C5B^{vii}	3.5920 (17)	H5B···H1B^{ix}	2.4200
C4···O32	3.0568 (17)	H5B···C11^{vii}	3.0900
C5A···C11^{vi}	3.3978 (17)	H5B···C10A^{vii}	3.0400
C5A···C10A^{vi}	3.5737 (18)	H6···H7^{viii}	2.5100
C5B···C3A^{vii}	3.5920 (17)	H7···C7^{viii}	3.0600
C5B···C3A^{vi}	3.5836 (17)	H7···C6^{viii}	3.0700
C5B···C3^{vi}	3.5874 (18)	H7···H6^{viii}	2.5100
C5B···C11^{vi}	3.5257 (18)	H7···H7^{viii}	2.4800
C6···N2^{vi}	3.3060 (19)	H8···O2^{xi}	2.6100
C7···C31^{vii}	3.598 (2)	H9···H9^{xii}	2.2400
C8···C31^{vi}	3.373 (2)	H10···C2^{i}	2.93 (2)
C9A···C3^{vi}	3.5846 (18)	H10···O2^{i}	1.92 (2)
C9A···C3A^{vi}	3.4443 (18)	H32A···O31	2.5800
C10A···C10A^{vi}	3.5439 (18)	H32A···O2^{ii}	2.7200
C10A···C5A^{vi}	3.5737 (18)	H32B···H4A^{ix}	2.5500
C11···C5B^{vi}	3.5257 (18)	H32C···O31	2.6600
C31—O32—C32	115.61 (11)	O31—C31—C3	124.08 (13)
C2—O3—H3	115 (2)	O32—C31—C3	111.81 (11)
N2—N1—C11	104.48 (11)	O31—C31—O32	124.11 (14)
N1—N2—C3	111.79 (10)	C3A—C4—H4A	109.00
C9A—N10—C10A	107.60 (11)	C3A—C4—H4B	109.00
N1—N2—H2	122.3 (16)	C5—C4—H4B	109.00
C3—N2—H2	125.5 (16)	H4A—C4—H4B	108.00

C9A—N10—H10	125.8 (14)	C5—C4—H4A	109.00
C10A—N10—H10	126.2 (14)	C4—C5—H5B	108.00
N2—C3—C31	119.39 (11)	C5A—C5—H5A	108.00
C3A—C3—C31	132.85 (12)	C5A—C5—H5B	108.00
N2—C3—C3A	107.76 (12)	H5A—C5—H5B	107.00
C3—C3A—C11	103.67 (11)	C4—C5—H5A	108.00
C4—C3A—C11	123.81 (11)	C5B—C6—H6	121.00
C3—C3A—C4	132.48 (13)	C7—C6—H6	121.00
C3A—C4—C5	114.64 (12)	C8—C7—H7	119.00
C4—C5—C5A	115.76 (10)	C6—C7—H7	119.00
C5—C5A—C5B	129.99 (11)	C9—C8—H8	119.00
C5—C5A—C10A	123.74 (12)	C7—C8—H8	119.00
C5B—C5A—C10A	106.14 (12)	C8—C9—H9	121.00
C6—C5B—C9A	118.86 (14)	C9A—C9—H9	121.00
C5A—C5B—C9A	106.79 (11)	O32—C32—H32A	109.00
C5A—C5B—C6	134.34 (14)	O32—C32—H32B	109.00
C5B—C6—C7	118.90 (14)	H32A—C32—H32B	109.00
C6—C7—C8	121.27 (14)	H32A—C32—H32C	109.00
C7—C8—C9	121.10 (17)	H32B—C32—H32C	109.00
C8—C9—C9A	117.89 (15)	O32—C32—H32C	109.00
N10—C9A—C9	129.59 (13)	O2—C2—C1	122.75 (15)
C5B—C9A—C9	121.97 (12)	O3—C2—C1	113.74 (12)
N10—C9A—C5B	108.44 (13)	O2—C2—O3	123.51 (14)
N10—C10A—C5A	111.01 (11)	C2—C1—H1A	109.00
N10—C10A—C11	127.96 (11)	C2—C1—H1B	109.00
C5A—C10A—C11	121.03 (13)	C2—C1—H1C	109.00
N1—C11—C3A	112.30 (11)	H1A—C1—H1B	109.00
C3A—C11—C10A	119.70 (10)	H1A—C1—H1C	109.00
N1—C11—C10A	128.01 (13)	H1B—C1—H1C	109.00
C32—O32—C31—O31	−0.8 (2)	C4—C5—C5A—C5B	−174.14 (12)
C32—O32—C31—C3	179.64 (13)	C4—C5—C5A—C10A	10.59 (18)
C11—N1—N2—C3	1.00 (14)	C10A—C5A—C5B—C6	178.22 (15)
N2—N1—C11—C3A	−1.05 (14)	C5—C5A—C5B—C9A	−176.44 (13)
N2—N1—C11—C10A	179.08 (12)	C5B—C5A—C10A—C11	−178.67 (11)
N1—N2—C3—C31	−179.77 (11)	C10A—C5A—C5B—C9A	−0.53 (14)
N1—N2—C3—C3A	−0.60 (15)	C5—C5A—C5B—C6	2.3 (2)
C9A—N10—C10A—C11	178.46 (12)	C5B—C5A—C10A—N10	1.18 (14)
C10A—N10—C9A—C5B	0.99 (15)	C5—C5A—C10A—N10	177.41 (12)
C9A—N10—C10A—C5A	−1.38 (15)	C5—C5A—C10A—C11	−2.44 (19)
C10A—N10—C9A—C9	−179.06 (14)	C9A—C5B—C6—C7	0.2 (2)
C31—C3—C3A—C4	1.4 (2)	C5A—C5B—C6—C7	−178.45 (14)
C31—C3—C3A—C11	178.96 (14)	C5A—C5B—C9A—N10	−0.29 (14)
N2—C3—C3A—C11	−0.06 (13)	C5A—C5B—C9A—C9	179.76 (13)
C3A—C3—C31—O32	2.4 (2)	C6—C5B—C9A—C9	0.8 (2)
N2—C3—C31—O31	1.8 (2)	C6—C5B—C9A—N10	−179.26 (12)
N2—C3—C3A—C4	−177.63 (13)	C5B—C6—C7—C8	−0.7 (2)
C3A—C3—C31—O31	−177.14 (14)	C6—C7—C8—C9	0.2 (2)
N2—C3—C31—O32	−178.66 (11)	C7—C8—C9—C9A	0.8 (2)
C3—C3A—C11—N1	0.70 (14)	C8—C9—C9A—N10	178.82 (14)

C3—C3A—C11—C10A	−179.41 (11)	C8—C9—C9A—C5B	−1.2 (2)
C11—C3A—C4—C5	9.58 (18)	N10—C10A—C11—N1	−2.5 (2)
C3—C3A—C4—C5	−173.26 (13)	C5A—C10A—C11—C3A	−2.53 (18)
C4—C3A—C11—N1	178.55 (11)	N10—C10A—C11—C3A	177.65 (12)
C4—C3A—C11—C10A	−1.57 (18)	C5A—C10A—C11—N1	177.33 (12)
C3A—C4—C5—C5A	−13.28 (16)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O31^{i}	0.90 (2)	1.95 (2)	2.8181 (14)	163 (2)
O3—H3···N1^{i}	0.97 (3)	1.76 (3)	2.7183 (16)	172 (3)
N10—H10···O2^{i}	0.93 (2)	1.92 (2)	2.8480 (17)	175 (2)

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1.

Fig. 1

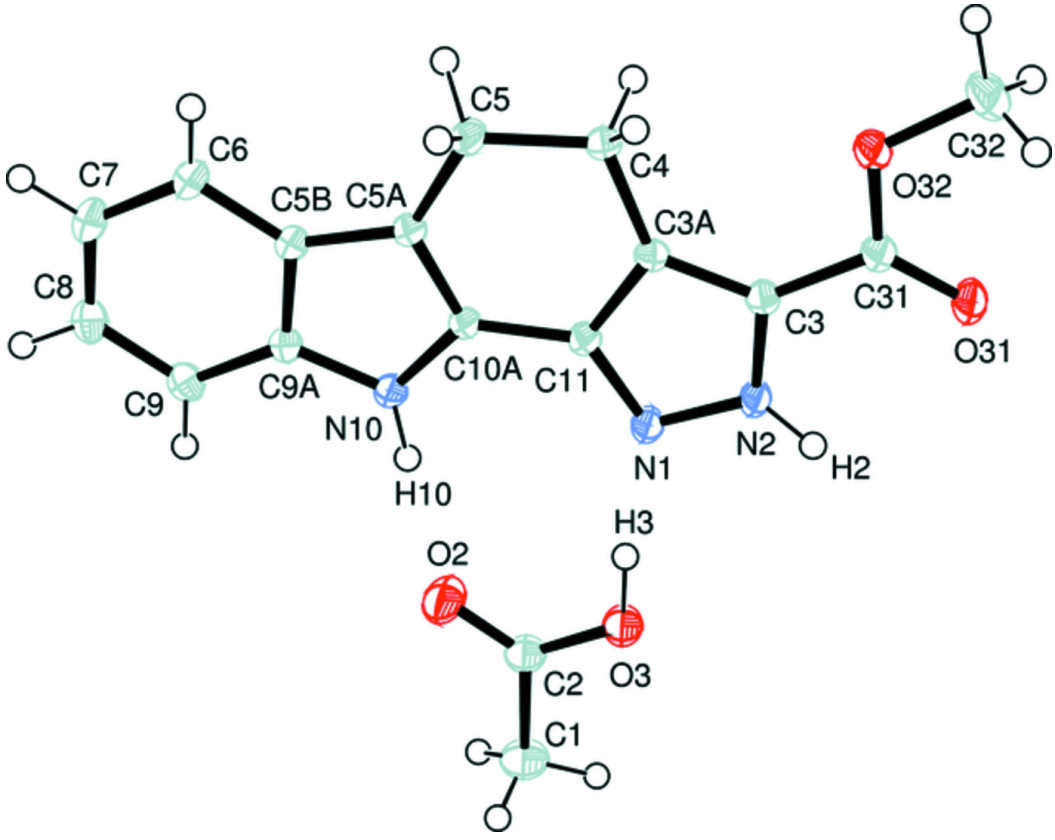
C5
C4
C6
C7
C5B
C5A
C3A
C32
O32
C8
C31
C3
O31
C10A
C11
C9A
C9
N10
N2
H2
H10
N1
H3
O2
C2
O3
C1

Fig. 2