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# 1,10-Dihydro-3,9-dimethylpyrazolo-[3,4-*a*]carbazole

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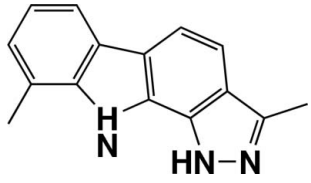
Key indicators: single-crystal X-ray study; $T$ = 203 K; mean $\sigma$(C–C) = 0.002 Å; disorder in main residue; $R$ factor = 0.043; $wR$ factor = 0.096; data-to-parameter ratio = 22.9.

The heterofused carbazole unit of the title compound, $C_{15}H_{13}N_3$, is planar. The planar pyrazole ring forms dihedral angles of 1.08 (7), 0.64 (6) and 2.60 (7)° with the pyrrole, fused benzene and methyl-substituted benzene rings, respectively. The methyl group on the five-membered ring is disordered equally over two positions. In the crystal structure, the molecules are stabilized by intermolecular N—H···N hydrogen bonds.

## Related literature

For related literature, see: Di Fabio *et al.* (2006); Gunaseelan *et al.* (2007*a*,*b*,*c*); Gunaseelan *et al.* (2007); Haider *et al.* (1998); Hedin *et al.* (2000); Hirata *et al.* (1999); Knolker & Reddy (2002); Thiruvalluvar *et al.* (2007).




## Experimental

### *Crystal data*

$C_{15}H_{13}N_3$
$M_r$ = 235.28
Monoclinic, $P2_1/c$
$a$ = 11.6553 (8) Å
$b$ = 5.7418 (3) Å
$c$ = 18.0804 (12) Å
$\beta$ = 101.796 (6)°
$V$ = 1184.43 (13) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 203 (2) K
0.51 × 0.43 × 0.37 mm

### *Data collection*

Oxford Diffraction Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
$T_{min}$ = 0.868, $T_{max}$ = 1.000
11247 measured reflections
3939 independent reflections
1587 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.047

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.096
$S$ = 0.81
3939 reflections
172 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···N2$^{i}$ | 0.906 (13) | 2.188 (12) | 2.9878 (14) | 147.0 (10) |
| N10—H10···N2$^{i}$ | 0.906 (13) | 2.263 (15) | 3.0558 (15) | 145.9 (12) |

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

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: BQ2027).

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o3471 [ **doi:10.1107/S160053680703351X** ]

# 1,10-Dihydro-3,9-dimethylpyrazolo[3,4-*a*]carbazole

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

## Comment

Our recent work on the *x*-ray structural studies of simple substituted carbazoles (Gunaseelan *et al.*, 2007 a, b & c; Thiruvalluvar *et al.*, 2007) directed us towards the structural studies of heterofused carbazoles (Gunaseelan *et al.*, 2007). The hetero-ring fused carbazoles have found an important place in the medicinal chemistry because of their high pharmacological properties (Hedin *et al.*, 2000, Hirata *et al.*, 1999, Haider *et al.*, 1998, Knolker & Reddy, 2002). The study of three dimensional arrangement of such compounds will provide some valuable information on synthetic as well as structure activity relationship (SAR) studies (Knolker & Reddy, 2002, Di Fabio *et al.*, 2006). In view of these discussions, here, we present the crystal structure of such a heterofused carbazole, the title compound, (I).

The molecular structure of (I), with atomic numbering scheme, is shown in Fig. 1. The heterofused carbazole unit is planar. The planar pyrazole ring forms dihedral angles of 1.08 (7)°, 0.64 (6)° and 2.60 (7)° with pyrrole, fused benzene and methyl substituted benzene ring, respectively. The methyl group at position 3 is disordered over two positions. In the crystal structure, the molecules are stabilized by intermolecular N—H···N ($-x + 1$, $y - 1/2$, $-z + 1/2$) hydrogen bonds (see Fig. 2).

## Experimental

Hydrazine hydrate (0.1 ml, 0.002 mol) was added to the solution of 2-acetyl-2,3,4,9-tetrahydro-1*H*-carbazol-1-one (240 mg, 0.001 mol) in glacial acetic acid (15 ml) and refluxed on oil bath for 1 h. The reaction was monitored by thin-layer chromatography. After the completion of the reaction it was poured into crushed ice. The precipitate was filtered, washed with water and dried to get the brown residue of crude compound (I), which was purified as a white powder by column chromatography over silica gel using petroleum ether-ethyl acetate (98:2 *v*/*v*) as eluant (50 mg, 20%) and recrystallized from glacial acetic.

## Refinement

H atoms bonded to N1 and N10 were 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 = 0.94–0.97 Å and $U_{iso}$(H) = 1.2–1.5 times $U_{eq}$(C). The methyl group at position 3 was found to be disordered over two positions.

## Figures

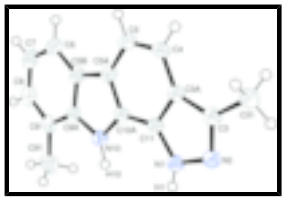


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

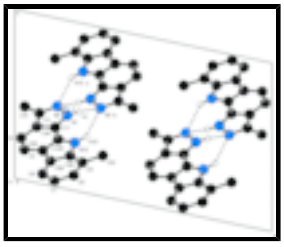


Fig. 2. The molecular packing of (I), viewed down the *b* axis. Dashed lines indicate hydrogen bonds. Symmetry code for both a and b: $-x + 1, y - 1/2, -z + 1/2$

### 1,10-Dihydro-3,9-dimethylpyrazolo[3,4-*a*]carbazole

*Crystal data*

| | |
|---|---|
| $C_{15}H_{13}N_3$ | $F_{000} = 496$ |
| $M_r = 235.28$ | $D_x = 1.319$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Melting point: 416(1) K |
| Hall symbol: -P 2ybc | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $a = 11.6553$ (8) Å | Cell parameters from 2712 reflections |
| $b = 5.7418$ (3) Å | $\theta = 4.8$–$32.4^\circ$ |
| $c = 18.0804$ (12) Å | $\mu = 0.08$ mm$^{-1}$ |
| $\beta = 101.796$ (6)° | $T = 203$ (2) K |
| $V = 1184.43$ (13) Å$^3$ | Plate, light_brown |
| $Z = 4$ | 0.51 × 0.43 × 0.37 mm |

*Data collection*

| | |
|---|---|
| Oxford Diffraction Gemini diffractometer | $R_{int} = 0.047$ |
| Radiation source: fine-focus sealed tube | $\theta_{max} = 32.4^\circ$ |
| Monochromator: graphite | $\theta_{min} = 4.8^\circ$ |
| $T = 203$(2) K | $h = -16 \rightarrow 12$ |
| φ and ω scans | $k = -8 \rightarrow 8$ |
| Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007) | $l = -26 \rightarrow 27$ |
| $T_{min} = 0.868$, $T_{max} = 1.000$ | Standard reflections: .; |
| 11247 measured reflections | every . reflections |
| 3939 independent reflections | intensity decay: . |
| 1587 reflections with $I > 2\sigma(I)$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.043$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.096$ | $w = 1/[\sigma^2(F_o^2) + (0.045P)^2]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |

| | |
|---|---|
| $S$ = 0.81 | $(\Delta/\sigma)_{max} < 0.001$ |
| 3939 reflections | $\Delta\rho_{max} = 0.18$ e Å$^{-3}$ |
| 172 parameters | $\Delta\rho_{min} = -0.21$ 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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| N1 | 0.43672 (9) | 0.39900 (18) | 0.20431 (6) | 0.0308 (4) | |
| N2 | 0.47899 (9) | 0.57711 (17) | 0.16614 (5) | 0.0313 (3) | |
| N10 | 0.28403 (9) | −0.04257 (18) | 0.23365 (6) | 0.0301 (3) | |
| C3 | 0.41609 (11) | 0.5733 (2) | 0.09612 (6) | 0.0289 (4) | |
| C3A | 0.33286 (10) | 0.3874 (2) | 0.08653 (6) | 0.0273 (4) | |
| C4 | 0.24616 (11) | 0.2997 (2) | 0.02588 (6) | 0.0321 (4) | |
| C5 | 0.18152 (11) | 0.1101 (2) | 0.03769 (6) | 0.0324 (4) | |
| C5A | 0.19858 (10) | 0.0022 (2) | 0.10985 (6) | 0.0283 (4) | |
| C5B | 0.14567 (10) | −0.1954 (2) | 0.13948 (6) | 0.0300 (4) | |
| C6 | 0.05963 (11) | −0.3574 (2) | 0.10872 (7) | 0.0377 (4) | |
| C7 | 0.03272 (12) | −0.5330 (2) | 0.15464 (8) | 0.0426 (5) | |
| C8 | 0.09011 (12) | −0.5508 (2) | 0.22997 (8) | 0.0417 (5) | |
| C9 | 0.17617 (11) | −0.3965 (2) | 0.26261 (7) | 0.0344 (4) | |
| C9A | 0.20113 (10) | −0.2182 (2) | 0.21583 (7) | 0.0291 (4) | |
| C10A | 0.28287 (10) | 0.0885 (2) | 0.16958 (6) | 0.0261 (4) | |
| C11 | 0.35017 (10) | 0.2806 (2) | 0.15764 (6) | 0.0263 (4) | |
| C31 | 0.43731 (12) | 0.7499 (2) | 0.03983 (7) | 0.0374 (4) | |
| C91 | 0.24276 (13) | −0.4178 (3) | 0.34279 (7) | 0.0503 (6) | |
| H1 | 0.4739 (12) | 0.358 (2) | 0.2515 (7) | 0.048 (4)* | |
| H4 | 0.23351 | 0.37154 | −0.02181 | 0.0385* | |
| H5 | 0.12498 | 0.05020 | −0.00244 | 0.0388* | |
| H6 | 0.02084 | −0.34717 | 0.05786 | 0.0452* | |
| H7 | −0.02538 | −0.64237 | 0.13466 | 0.0510* | |
| H8 | 0.06942 | −0.67235 | 0.25954 | 0.0500* | |
| H10 | 0.3347 (13) | −0.015 (2) | 0.2779 (7) | 0.053 (4)* | |
| H31A | 0.49975 | 0.85390 | 0.06335 | 0.0560* | 0.500 |
| H31B | 0.36626 | 0.83888 | 0.02215 | 0.0560* | 0.500 |
| H31C | 0.45969 | 0.67151 | −0.00261 | 0.0560* | 0.500 |

| | | | | | |
|---|---|---|---|---|---|
| H31D | 0.38405 | 0.72230 | −0.00809 | 0.0560* | 0.500 |
| H31E | 0.51754 | 0.73732 | 0.03311 | 0.0560* | 0.500 |
| H31F | 0.42411 | 0.90468 | 0.05787 | 0.0560* | 0.500 |
| H91A | 0.21018 | −0.54402 | 0.36762 | 0.0754* | |
| H91B | 0.23653 | −0.27322 | 0.36946 | 0.0754* | |
| H91C | 0.32456 | −0.44983 | 0.34305 | 0.0754* | |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0319 (6) | 0.0337 (7) | 0.0245 (6) | −0.0023 (5) | 0.0006 (5) | 0.0018 (5) |
| N2 | 0.0321 (6) | 0.0309 (6) | 0.0301 (6) | −0.0016 (5) | 0.0043 (5) | 0.0025 (5) |
| N10 | 0.0270 (6) | 0.0348 (6) | 0.0274 (6) | −0.0005 (5) | 0.0027 (5) | 0.0021 (5) |
| C3 | 0.0299 (7) | 0.0297 (7) | 0.0273 (7) | 0.0069 (6) | 0.0060 (5) | 0.0003 (6) |
| C3A | 0.0276 (7) | 0.0287 (7) | 0.0259 (6) | 0.0058 (6) | 0.0059 (5) | −0.0022 (5) |
| C4 | 0.0340 (7) | 0.0383 (8) | 0.0221 (6) | 0.0067 (7) | 0.0016 (5) | −0.0004 (6) |
| C5 | 0.0293 (7) | 0.0367 (8) | 0.0283 (7) | 0.0027 (6) | −0.0008 (5) | −0.0057 (6) |
| C5A | 0.0258 (7) | 0.0289 (7) | 0.0291 (7) | 0.0049 (6) | 0.0029 (5) | −0.0031 (6) |
| C5B | 0.0244 (7) | 0.0298 (7) | 0.0360 (7) | 0.0024 (6) | 0.0066 (5) | −0.0049 (6) |
| C6 | 0.0289 (7) | 0.0399 (8) | 0.0426 (8) | 0.0008 (6) | 0.0037 (6) | −0.0087 (7) |
| C7 | 0.0336 (8) | 0.0353 (8) | 0.0606 (10) | −0.0067 (7) | 0.0139 (7) | −0.0103 (7) |
| C8 | 0.0370 (8) | 0.0338 (8) | 0.0595 (10) | 0.0011 (7) | 0.0219 (7) | 0.0033 (7) |
| C9 | 0.0313 (7) | 0.0329 (8) | 0.0419 (8) | 0.0032 (6) | 0.0143 (6) | 0.0029 (6) |
| C9A | 0.0253 (7) | 0.0289 (7) | 0.0340 (7) | 0.0035 (6) | 0.0083 (5) | −0.0020 (6) |
| C10A | 0.0269 (7) | 0.0273 (7) | 0.0241 (6) | 0.0048 (6) | 0.0049 (5) | −0.0001 (5) |
| C11 | 0.0246 (6) | 0.0288 (7) | 0.0245 (6) | 0.0035 (6) | 0.0024 (5) | −0.0033 (6) |
| C31 | 0.0419 (8) | 0.0352 (8) | 0.0359 (7) | 0.0020 (7) | 0.0100 (6) | 0.0042 (6) |
| C91 | 0.0505 (10) | 0.0557 (10) | 0.0467 (9) | 0.0045 (8) | 0.0147 (7) | 0.0151 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| N1—N2 | 1.3798 (14) | C8—C9 | 1.3774 (18) |
| N1—C11 | 1.3579 (16) | C9—C9A | 1.3960 (17) |
| N2—C3 | 1.3271 (14) | C9—C91 | 1.5030 (18) |
| N10—C9A | 1.3882 (16) | C10A—C11 | 1.3956 (16) |
| N10—C10A | 1.3792 (15) | C4—H4 | 0.9400 |
| N1—H1 | 0.906 (13) | C5—H5 | 0.9400 |
| N10—H10 | 0.906 (13) | C6—H6 | 0.9400 |
| C3—C3A | 1.4289 (17) | C7—H7 | 0.9400 |
| C3—C31 | 1.4927 (17) | C8—H8 | 0.9400 |
| C3A—C4 | 1.4227 (16) | C31—H31A | 0.9700 |
| C3A—C11 | 1.4013 (15) | C31—H31B | 0.9700 |
| C4—C5 | 1.3653 (17) | C31—H31C | 0.9700 |
| C5—C5A | 1.4211 (15) | C31—H31D | 0.9700 |
| C5A—C5B | 1.4456 (16) | C31—H31E | 0.9700 |
| C5A—C10A | 1.3940 (16) | C31—H31F | 0.9700 |
| C5B—C6 | 1.3972 (17) | C91—H91A | 0.9700 |
| C5B—C9A | 1.4056 (16) | C91—H91B | 0.9700 |
| C6—C7 | 1.3823 (18) | C91—H91C | 0.9700 |

| | | | |
|---|---|---|---|
| C7—C8 | 1.393 (2) | | |
| N1···N10 | 3.2036 (15) | C10A···H31F$^{iv}$ | 3.0400 |
| N1···N2$^{i}$ | 2.9878 (14) | C11···H31F$^{iv}$ | 3.0500 |
| N1···N10$^{ii}$ | 3.2360 (15) | C31···H31C$^{v}$ | 2.8400 |
| N2···N10$^{ii}$ | 3.0558 (15) | C91···H10 | 2.896 (13) |
| N2···N1$^{ii}$ | 2.9878 (14) | C91···H7$^{vii}$ | 3.0800 |
| N10···N1$^{i}$ | 3.2360 (15) | C91···H31D$^{viii}$ | 3.0700 |
| N10···N1 | 3.2036 (15) | H1···N1$^{i}$ | 2.886 (12) |
| N10···N2$^{i}$ | 3.0558 (15) | H1···N2$^{i}$ | 2.188 (12) |
| N1···H10$^{ii}$ | 2.664 (15) | H1···N10$^{ii}$ | 2.837 (14) |
| N1···H1$^{ii}$ | 2.886 (12) | H1···H10$^{ii}$ | 2.51 (2) |
| N2···H1$^{ii}$ | 2.188 (12) | H5···H6$^{vi}$ | 2.4700 |
| N2···H10$^{ii}$ | 2.263 (15) | H6···C5$^{vi}$ | 2.9600 |
| N10···H91B | 2.9400 | H6···H5$^{vi}$ | 2.4700 |
| N10···H1$^{i}$ | 2.837 (14) | H7···C91$^{ix}$ | 3.0800 |
| C3···C5A$^{iii}$ | 3.5792 (17) | H7···H91B$^{ix}$ | 2.5600 |
| C3A···C5B$^{iii}$ | 3.5027 (17) | H8···H91A | 2.4000 |
| C4···C6$^{iii}$ | 3.4897 (18) | H8···C7$^{ix}$ | 2.9800 |
| C5A···C31$^{iv}$ | 3.5894 (18) | H8···C8$^{ix}$ | 2.8900 |
| C5A···C7$^{iii}$ | 3.4865 (17) | H8···C9$^{ix}$ | 3.0900 |
| C5A···C3$^{iv}$ | 3.5792 (17) | H10···C91 | 2.896 (13) |
| C5B···C3A$^{iv}$ | 3.5027 (17) | H10···N1$^{i}$ | 2.664 (15) |
| C6···C4$^{iv}$ | 3.4897 (18) | H10···N2$^{i}$ | 2.263 (15) |
| C7···C5A$^{iv}$ | 3.4865 (17) | H10···H1$^{i}$ | 2.51 (2) |
| C8···C10A$^{iv}$ | 3.3959 (18) | H31B···C4$^{iii}$ | 3.0000 |
| C9···C11$^{iv}$ | 3.5674 (17) | H31B···C5$^{iii}$ | 2.7200 |
| C10A···C8$^{iii}$ | 3.3959 (18) | H31B···C5A$^{iii}$ | 2.9100 |
| C11···C9$^{iii}$ | 3.5674 (17) | H31C···C3$^{v}$ | 2.8200 |
| C31···C5A$^{iii}$ | 3.5894 (18) | H31C···C31$^{v}$ | 2.8400 |
| C3···H31C$^{v}$ | 2.8200 | H31D···C4 | 3.0400 |
| C3A···H31F$^{iv}$ | 3.0500 | H31D···C91$^{x}$ | 3.0700 |
| C4···H31F$^{iv}$ | 3.0500 | H31D···H91B$^{x}$ | 2.5300 |
| C4···H31B$^{iv}$ | 3.0000 | H31F···C3A$^{iii}$ | 3.0500 |
| C4···H31D | 3.0400 | H31F···C4$^{iii}$ | 3.0500 |
| C5···H31B$^{iv}$ | 2.7200 | H31F···C5$^{iii}$ | 3.0200 |
| C5···H6$^{vi}$ | 2.9600 | H31F···C5A$^{iii}$ | 3.0200 |
| C5···H31F$^{iv}$ | 3.0200 | H31F···C10A$^{iii}$ | 3.0400 |
| C5A···H31F$^{iv}$ | 3.0200 | H31F···C11$^{iii}$ | 3.0500 |
| C5A···H31B$^{iv}$ | 2.9100 | H91A···H8 | 2.4000 |
| C7···H8$^{vii}$ | 2.9800 | H91B···N10 | 2.9400 |
| C8···H8$^{vii}$ | 2.8900 | H91B···H7$^{vii}$ | 2.5600 |

| | | | |
|---|---|---|---|
| C9···H8$^{vii}$ | 3.0900 | H91B···H31D$^{viii}$ | 2.5300 |
| N2—N1—C11 | 110.86 (10) | C5—C4—H4 | 120.00 |
| N1—N2—C3 | 106.31 (10) | C4—C5—H5 | 120.00 |
| C9A—N10—C10A | 108.34 (10) | C5A—C5—H5 | 120.00 |
| C11—N1—H1 | 127.4 (8) | C5B—C6—H6 | 121.00 |
| N2—N1—H1 | 120.9 (8) | C7—C6—H6 | 121.00 |
| C9A—N10—H10 | 129.0 (8) | C6—C7—H7 | 119.00 |
| C10A—N10—H10 | 122.6 (8) | C8—C7—H7 | 119.00 |
| N2—C3—C31 | 120.20 (11) | C7—C8—H8 | 119.00 |
| C3A—C3—C31 | 128.88 (10) | C9—C8—H8 | 119.00 |
| N2—C3—C3A | 110.92 (10) | C3—C31—H31A | 109.00 |
| C3—C3A—C11 | 104.40 (10) | C3—C31—H31B | 109.00 |
| C4—C3A—C11 | 119.91 (11) | C3—C31—H31C | 109.00 |
| C3—C3A—C4 | 135.69 (10) | C3—C31—H31D | 109.00 |
| C3A—C4—C5 | 119.30 (10) | C3—C31—H31E | 109.00 |
| C4—C5—C5A | 120.87 (10) | C3—C31—H31F | 109.00 |
| C5—C5A—C5B | 133.80 (11) | H31A—C31—H31B | 109.00 |
| C5—C5A—C10A | 120.03 (11) | H31A—C31—H31C | 109.00 |
| C5B—C5A—C10A | 106.16 (9) | H31A—C31—H31D | 141.00 |
| C5A—C5B—C9A | 106.96 (10) | H31A—C31—H31E | 56.00 |
| C6—C5B—C9A | 118.52 (11) | H31A—C31—H31F | 56.00 |
| C5A—C5B—C6 | 134.50 (10) | H31B—C31—H31C | 109.00 |
| C5B—C6—C7 | 118.62 (11) | H31B—C31—H31D | 56.00 |
| C6—C7—C8 | 121.21 (12) | H31B—C31—H31E | 141.00 |
| C7—C8—C9 | 122.26 (12) | H31B—C31—H31F | 56.00 |
| C8—C9—C91 | 123.24 (12) | H31C—C31—H31D | 56.00 |
| C9A—C9—C91 | 120.90 (12) | H31C—C31—H31E | 56.00 |
| C8—C9—C9A | 115.84 (12) | H31C—C31—H31F | 141.00 |
| N10—C9A—C5B | 108.59 (10) | H31D—C31—H31E | 109.00 |
| N10—C9A—C9 | 127.84 (11) | H31D—C31—H31F | 109.00 |
| C5B—C9A—C9 | 123.53 (11) | H31E—C31—H31F | 109.00 |
| N10—C10A—C5A | 109.94 (10) | C9—C91—H91A | 109.00 |
| N10—C10A—C11 | 130.68 (10) | C9—C91—H91B | 109.00 |
| C5A—C10A—C11 | 119.37 (10) | C9—C91—H91C | 109.00 |
| N1—C11—C10A | 132.01 (10) | H91A—C91—H91B | 109.00 |
| C3A—C11—C10A | 120.51 (10) | H91A—C91—H91C | 109.00 |
| N1—C11—C3A | 107.48 (10) | H91B—C91—H91C | 109.00 |
| C3A—C4—H4 | 120.00 | | |
| C11—N1—N2—C3 | 1.77 (13) | C10A—C5A—C5B—C6 | −178.14 (13) |
| N2—N1—C11—C3A | −1.32 (13) | C10A—C5A—C5B—C9A | −0.06 (14) |
| N2—N1—C11—C10A | 179.04 (12) | C5—C5A—C10A—N10 | −179.96 (11) |
| N1—N2—C3—C3A | −1.52 (13) | C5—C5A—C10A—C11 | −0.21 (17) |
| N1—N2—C3—C31 | 178.28 (11) | C5B—C5A—C10A—N10 | −0.44 (13) |
| C10A—N10—C9A—C5B | −0.82 (13) | C5B—C5A—C10A—C11 | 179.31 (11) |
| C10A—N10—C9A—C9 | 177.07 (12) | C5A—C5B—C6—C7 | 177.92 (13) |
| C9A—N10—C10A—C5A | 0.79 (14) | C9A—C5B—C6—C7 | 0.00 (19) |
| C9A—N10—C10A—C11 | −178.93 (12) | C5A—C5B—C9A—N10 | 0.54 (13) |
| N2—C3—C3A—C4 | −179.12 (13) | C5A—C5B—C9A—C9 | −177.46 (11) |

| | | | |
|---|---|---|---|
| N2—C3—C3A—C11 | 0.74 (14) | C6—C5B—C9A—N10 | 178.97 (11) |
| C31—C3—C3A—C4 | 1.1 (2) | C6—C5B—C9A—C9 | 0.97 (18) |
| C31—C3—C3A—C11 | −179.03 (12) | C5B—C6—C7—C8 | −0.41 (19) |
| C3—C3A—C4—C5 | 179.16 (13) | C6—C7—C8—C9 | −0.1 (2) |
| C11—C3A—C4—C5 | −0.69 (18) | C7—C8—C9—C9A | 1.05 (19) |
| C3—C3A—C11—N1 | 0.36 (13) | C7—C8—C9—C91 | −177.40 (13) |
| C3—C3A—C11—C10A | −179.94 (12) | C8—C9—C9A—N10 | −179.07 (12) |
| C4—C3A—C11—N1 | −179.75 (11) | C8—C9—C9A—C5B | −1.48 (18) |
| C4—C3A—C11—C10A | −0.06 (18) | C91—C9—C9A—N10 | −0.6 (2) |
| C3A—C4—C5—C5A | 0.98 (18) | C91—C9—C9A—C5B | 177.01 (12) |
| C4—C5—C5A—C5B | −179.90 (13) | N10—C10A—C11—N1 | −0.2 (2) |
| C4—C5—C5A—C10A | −0.54 (18) | N10—C10A—C11—C3A | −179.81 (12) |
| C5—C5A—C5B—C6 | 1.3 (2) | C5A—C10A—C11—N1 | −179.90 (12) |
| C5—C5A—C5B—C9A | 179.36 (13) | C5A—C10A—C11—C3A | 0.49 (17) |

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

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···N2$^{i}$ | 0.906 (13) | 2.188 (12) | 2.9878 (14) | 147.0 (10) |
| N10—H10···N2$^{i}$ | 0.906 (13) | 2.263 (15) | 3.0558 (15) | 145.9 (12) |

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

**Fig. 1**

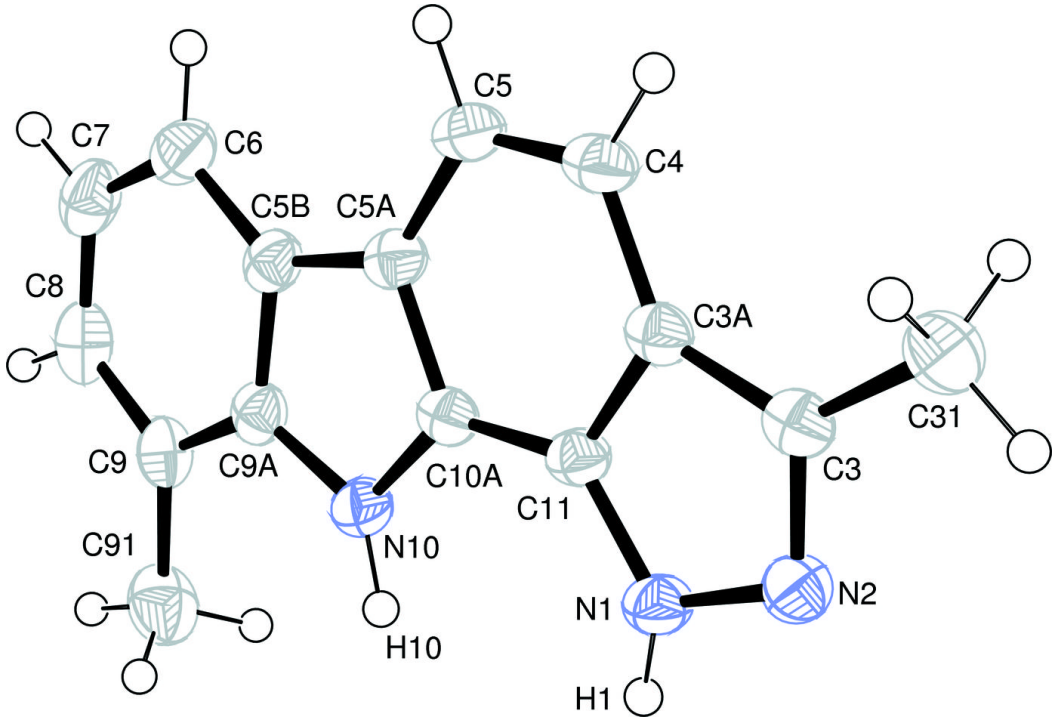
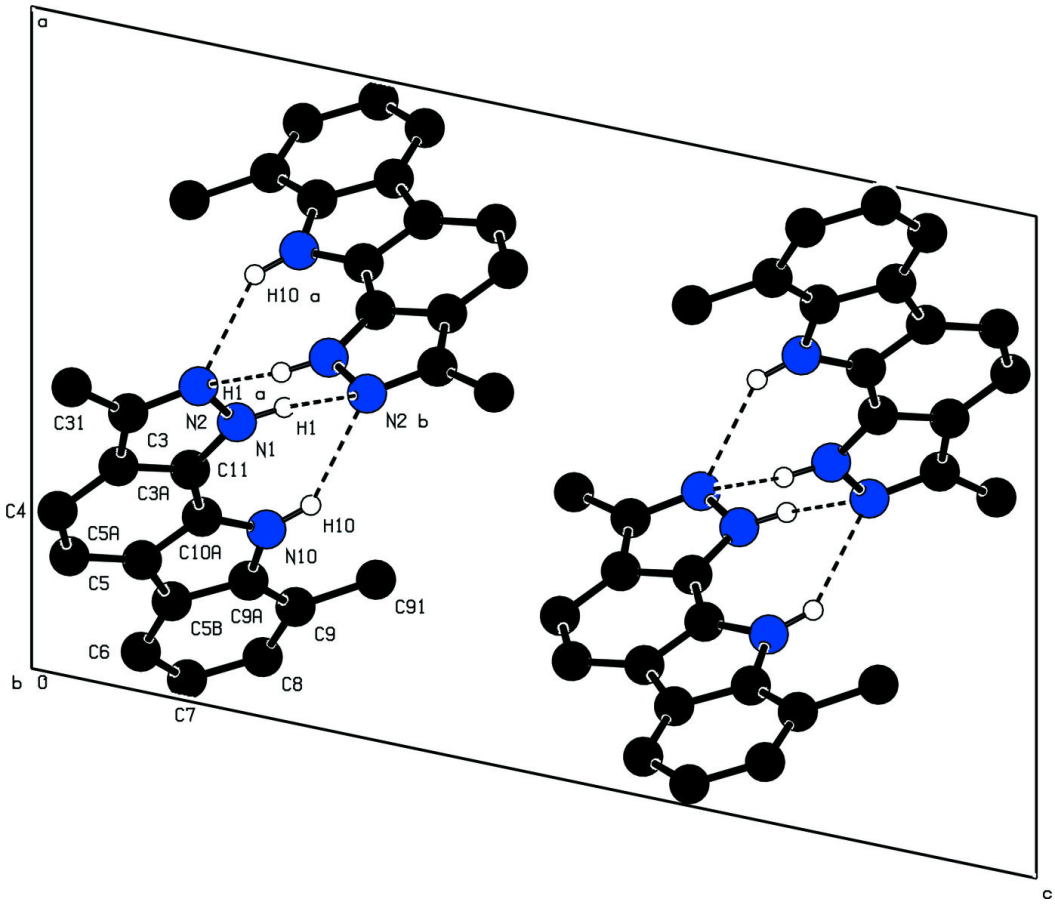

C5
C7
C6
C4
C5B
C5A
C8
C3A
C31
C9
C9A
C10A
C11
C3
C91
N10
N1
N2
H10
H1

**Fig. 2**


a
H10 a
H1 a
C31
N2
C3
N1
H1
N2 b
C11
C3A
C4
C5A
C10A
H10
N10
C5
C9A
C91
C5B
C9
C6
C8
b 0
C7
c