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5-Methyl-7,8,9,10-tetrahydrocyclohepta[*b*]indol-6(5*H*)-one

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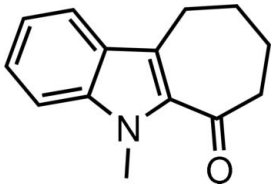
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.106; data-to-parameter ratio = 8.1.

In the title molecule, $C_{14}H_{15}NO$, the dihedral angle between the benzene and pyrrole rings is 1.99 (12)°. The cycloheptene ring adopts a slightly distorted boat conformation.

Related literature

For the interest and importance of indole derivatives, see: Csomós *et al.* (2007). For pyrido-fused cyclohept[*b*]indole alkaloids, see: Bennasar *et al.* (1997). For crystallographic studies of cyclohept[*b*]indoles, see: Archana *et al.* (2010).



Experimental

Crystal data

$C_{14}H_{15}NO$ M_r = 213.27
Orthorhombic, $Pca2_1$
a = 8.6999 (2) Å
b = 14.1805 (3) Å
c = 9.1392 (3) Å
V = 1127.49 (5) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.62 mm^{-1}
T = 295 K
0.47 × 0.35 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur Ruby Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
T_{min} = 0.803, T_{max} = 1.000
1184 measured reflections
1184 independent reflections
1148 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.106
S = 1.07
1184 reflections
147 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5032).

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supporting information

Acta Cryst. (2011). E**67**, o1325 [doi:10.1107/S1600536811016229]

5-Methyl-7,8,9,10-tetrahydrocyclohepta[*b*]indol-6(5*H*)-one

R. Archana, E. Yamuna, K. J. Rajendra Prasad, A. Thiruvalluvar and R. J. Butcher

S1. Comment

Indole derivatives condensed with different heterocycles are physiologically active compounds found in abundance in materials such as pharmaceuticals, alkaloids and potential therapeutic agents (Csomós *et al.*, 2007). Ervitsine and Ervatamine (Bennasar *et al.*, 1997) were important class of pyrido fused cyclohept[*b*]indole alkaloids. Recently we have reported crystallographic studies for some cyclohept[*b*]indoles in our laboratory (Archana *et al.*, 2010).

The molecular structure of the title compound, with atomic numbering scheme, is shown in Fig. 1. In the title molecule, $C_{14}H_{15}NO$, the dihedral angle between the benzene and pyrrole rings is 1.99 (12)°. The cycloheptene ring adopts a slightly distorted boat conformation.

S2. Experimental

To a solution of 7,8,9,10-tetrahydrocyclohepta[*b*]indol-6(5*H*)-one (0.199 g, 0.001 mol) in 5 ml acetone added powdered KOH (0.280 g, 0.005 mol) in ice cold condition. After few minutes methyl iodide (0.13 ml, 0.002 mol) was added drop by drop with vigorous stirring and the reaction mixture was stirrired for 15 min at room temperature. Benzene was added to the reaction mixture and insoluble materials are removed by filtration. The benzene solution was washed with saturated NaCl solution, dried by using Na_2SO_4 and evaporation yielded the title compound (0.191 g, 90%). This was recrystallized from benzene and ethyl acetate mixture.

S3. Refinement

Owing to the absence of any anamalous scatterers in the molecule, the Friedel pairs were merged. The absolute structure in the present model have been chosen arbitrarily. H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93 - 0.97 Å and U_{iso}(H) = 1.2 - 1.5 times U_{eq}(C).

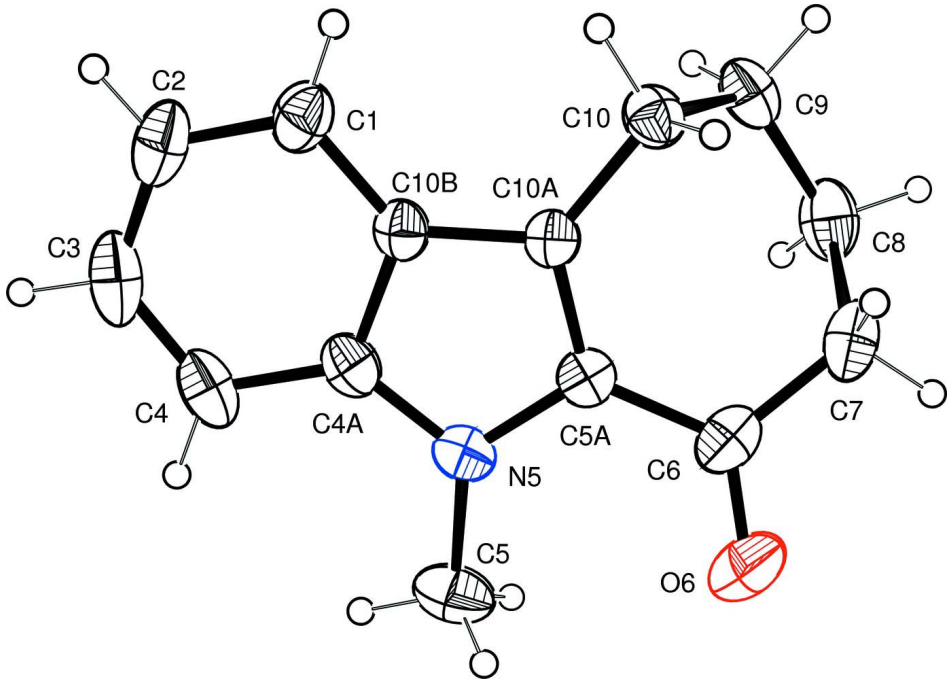

Figure 1

The molecular structure of the title compound, showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level. H atoms are shown as small spheres of arbitrary radius.

5-Methyl-7,8,9,10-tetrahydrocyclohepta[*b*]indol-6(5*H*)-one

Crystal data

$C_{14}H_{15}NO$
$M_r = 213.27$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
$a = 8.6999$ (2) Å
$b = 14.1805$ (3) Å
$c = 9.1392$ (3) Å
$V = 1127.49$ (5) Å^3
$Z = 4$
$F(000) = 456$
$D_x = 1.256$ Mg m^{-3}
Melting point: 338 K
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 2006 reflections
$\theta = 4.8$–$73.4°$
$\mu = 0.62$ mm^{-1}
$T = 295$ K
Chunk, pale-yellow
0.47 × 0.35 × 0.20 mm

Data collection

Oxford Diffraction Xcalibur Ruby Gemini diffractometer
Radiation source: Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 10.5081 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
$T_{min} = 0.803$, $T_{max} = 1.000$
1184 measured reflections
1184 independent reflections
1148 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 73.6°$, $\theta_{min} = 6.0°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.106$
$S = 1.07$
1184 reflections
147 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0678P)^2 + 0.0651P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.018 (2)
Absolute structure: see *Refinement* section in *Supplementary materials*

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 $F^2 > 2\sigma(F^2)$ 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}
O6	0.5677 (3)	0.05292 (17)	0.4636 (4)	0.1141 (10)
N5	0.4250 (2)	0.18603 (13)	0.6610 (2)	0.0514 (5)
C1	0.4932 (3)	0.42768 (16)	0.7515 (3)	0.0607 (8)
C2	0.3719 (3)	0.4495 (2)	0.8403 (4)	0.0792 (10)
C3	0.2600 (4)	0.3822 (2)	0.8755 (4)	0.0826 (10)
C4	0.2661 (3)	0.2923 (2)	0.8220 (3)	0.0686 (9)
C4A	0.3895 (2)	0.26879 (16)	0.7301 (2)	0.0512 (7)
C5	0.3392 (3)	0.09825 (18)	0.6805 (4)	0.0791 (10)
C5A	0.5637 (3)	0.19791 (14)	0.5882 (2)	0.0471 (6)
C6	0.6360 (4)	0.12408 (16)	0.4995 (3)	0.0637 (9)
C7	0.7989 (3)	0.13870 (18)	0.4529 (3)	0.0669 (9)
C8	0.9072 (3)	0.1731 (2)	0.5724 (3)	0.0713 (9)
C9	0.9058 (3)	0.2786 (2)	0.6046 (3)	0.0647 (8)
C10	0.7621 (3)	0.33087 (14)	0.5528 (3)	0.0533 (6)
C10A	0.6154 (2)	0.28910 (14)	0.6081 (2)	0.0431 (5)
C10B	0.5048 (2)	0.33542 (14)	0.6960 (2)	0.0458 (6)
H1	0.56661	0.47293	0.72821	0.0728*
H2	0.36341	0.51021	0.87808	0.0950*
H3	0.17945	0.39907	0.93704	0.0989*
H4	0.19098	0.24819	0.84567	0.0823*
H5A	0.25845	0.10790	0.75052	0.1184*
H5B	0.29541	0.07936	0.58858	0.1184*
H5C	0.40722	0.04986	0.71507	0.1184*
H7A	0.83814	0.07958	0.41476	0.0803*

H7B	0.80020	0.18409	0.37351	0.0803*
H8A	0.88223	0.13988	0.66208	0.0856*
H8B	1.01113	0.15535	0.54543	0.0856*
H9A	0.91596	0.28753	0.70940	0.0776*
H9B	0.99487	0.30708	0.55857	0.0776*
H10A	0.76018	0.33072	0.44669	0.0640*
H10B	0.76828	0.39599	0.58476	0.0640*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O6	0.131 (2)	0.0782 (13)	0.133 (2)	−0.0248 (14)	0.030 (2)	−0.0505 (16)
N5	0.0472 (9)	0.0535 (9)	0.0534 (10)	−0.0053 (7)	−0.0045 (8)	0.0057 (8)
C1	0.0620 (13)	0.0556 (12)	0.0644 (14)	0.0153 (10)	−0.0049 (12)	−0.0035 (11)
C2	0.0837 (19)	0.0764 (16)	0.0774 (18)	0.0354 (15)	0.0030 (16)	−0.0123 (16)
C3	0.0671 (15)	0.111 (2)	0.0696 (17)	0.0405 (18)	0.0133 (15)	0.0049 (17)
C4	0.0442 (12)	0.0964 (17)	0.0652 (15)	0.0115 (12)	0.0043 (11)	0.0162 (15)
C4A	0.0408 (10)	0.0643 (12)	0.0486 (12)	0.0048 (9)	−0.0069 (9)	0.0089 (10)
C5	0.0732 (17)	0.0691 (14)	0.095 (2)	−0.0234 (13)	−0.0062 (17)	0.0152 (16)
C5A	0.0508 (11)	0.0484 (10)	0.0421 (10)	0.0017 (8)	−0.0035 (9)	0.0048 (9)
C6	0.0855 (18)	0.0514 (12)	0.0541 (14)	0.0054 (11)	−0.0011 (13)	−0.0046 (10)
C7	0.0838 (18)	0.0654 (13)	0.0514 (13)	0.0252 (12)	0.0133 (13)	0.0021 (11)
C8	0.0639 (14)	0.0833 (17)	0.0668 (16)	0.0278 (13)	0.0048 (13)	0.0131 (15)
C9	0.0421 (11)	0.0857 (16)	0.0663 (15)	0.0022 (11)	0.0033 (11)	0.0069 (14)
C10	0.0538 (11)	0.0519 (9)	0.0542 (13)	−0.0005 (9)	0.0045 (10)	0.0087 (9)
C10A	0.0436 (10)	0.0438 (8)	0.0418 (10)	0.0050 (7)	−0.0036 (8)	0.0043 (8)
C10B	0.0421 (10)	0.0506 (10)	0.0448 (11)	0.0083 (7)	−0.0055 (8)	0.0029 (8)

Geometric parameters (Å, º)

O6—C6	1.216 (4)	C10A—C10B	1.415 (3)
N5—C4A	1.368 (3)	C1—H1	0.9300
N5—C5	1.462 (3)	C2—H2	0.9300
N5—C5A	1.388 (3)	C3—H3	0.9300
C1—C2	1.367 (4)	C4—H4	0.9300
C1—C10B	1.407 (3)	C5—H5A	0.9600
C2—C3	1.401 (4)	C5—H5B	0.9600
C3—C4	1.366 (4)	C5—H5C	0.9600
C4—C4A	1.403 (3)	C7—H7A	0.9700
C4A—C10B	1.413 (3)	C7—H7B	0.9700
C5A—C6	1.466 (3)	C8—H8A	0.9700
C5A—C10A	1.381 (3)	C8—H8B	0.9700
C6—C7	1.494 (4)	C9—H9A	0.9700
C7—C8	1.523 (4)	C9—H9B	0.9700
C8—C9	1.525 (4)	C10—H10A	0.9700
C9—C10	1.529 (4)	C10—H10B	0.9700
C10—C10A	1.495 (3)		

C4A—N5—C5	123.97 (19)	C2—C3—H3	119.00
C4A—N5—C5A	108.26 (17)	C4—C3—H3	119.00
C5—N5—C5A	127.26 (19)	C3—C4—H4	121.00
C2—C1—C10B	118.7 (2)	C4A—C4—H4	121.00
C1—C2—C3	121.3 (3)	N5—C5—H5A	109.00
C2—C3—C4	121.8 (3)	N5—C5—H5B	109.00
C3—C4—C4A	117.8 (3)	N5—C5—H5C	110.00
N5—C4A—C4	130.8 (2)	H5A—C5—H5B	109.00
N5—C4A—C10B	108.16 (16)	H5A—C5—H5C	109.00
C4—C4A—C10B	121.1 (2)	H5B—C5—H5C	109.00
N5—C5A—C6	123.5 (2)	C6—C7—H7A	108.00
N5—C5A—C10A	109.49 (18)	C6—C7—H7B	108.00
C6—C5A—C10A	127.0 (2)	C8—C7—H7A	108.00
O6—C6—C5A	122.2 (3)	C8—C7—H7B	108.00
O6—C6—C7	120.1 (3)	H7A—C7—H7B	107.00
C5A—C6—C7	117.7 (2)	C7—C8—H8A	108.00
C6—C7—C8	115.3 (2)	C7—C8—H8B	108.00
C7—C8—C9	116.6 (2)	C9—C8—H8A	108.00
C8—C9—C10	115.0 (2)	C9—C8—H8B	108.00
C9—C10—C10A	113.67 (19)	H8A—C8—H8B	107.00
C5A—C10A—C10	127.19 (19)	C8—C9—H9A	109.00
C5A—C10A—C10B	106.73 (17)	C8—C9—H9B	109.00
C10—C10A—C10B	126.05 (18)	C10—C9—H9A	108.00
C1—C10B—C4A	119.44 (18)	C10—C9—H9B	108.00
C1—C10B—C10A	133.24 (19)	H9A—C9—H9B	108.00
C4A—C10B—C10A	107.31 (17)	C9—C10—H10A	109.00
C2—C1—H1	121.00	C9—C10—H10B	109.00
C10B—C1—H1	121.00	C10A—C10—H10A	109.00
C1—C2—H2	119.00	C10A—C10—H10B	109.00
C3—C2—H2	119.00	H10A—C10—H10B	108.00

C5—N5—C4A—C4	−4.8 (4)	N5—C5A—C6—O6	12.5 (4)
C5—N5—C4A—C10B	174.5 (2)	N5—C5A—C6—C7	−167.6 (2)
C5A—N5—C4A—C4	−177.2 (2)	C10A—C5A—C6—O6	−164.4 (3)
C5A—N5—C4A—C10B	2.2 (2)	C10A—C5A—C6—C7	15.4 (4)
C4A—N5—C5A—C6	−178.7 (2)	N5—C5A—C10A—C10	177.9 (2)
C4A—N5—C5A—C10A	−1.3 (2)	N5—C5A—C10A—C10B	−0.1 (2)
C5—N5—C5A—C6	9.3 (4)	C6—C5A—C10A—C10	−4.8 (4)
C5—N5—C5A—C10A	−173.3 (2)	C6—C5A—C10A—C10B	177.2 (2)
C10B—C1—C2—C3	−0.4 (5)	O6—C6—C7—C8	−134.4 (3)
C2—C1—C10B—C4A	1.6 (3)	C5A—C6—C7—C8	45.7 (3)
C2—C1—C10B—C10A	−177.2 (2)	C6—C7—C8—C9	−81.6 (3)
C1—C2—C3—C4	−0.6 (5)	C7—C8—C9—C10	19.9 (3)
C2—C3—C4—C4A	0.3 (5)	C8—C9—C10—C10A	54.1 (3)
C3—C4—C4A—N5	−179.7 (3)	C9—C10—C10A—C5A	−57.7 (3)
C3—C4—C4A—C10B	1.0 (4)	C9—C10—C10A—C10B	119.9 (2)
N5—C4A—C10B—C1	178.60 (19)	C5A—C10A—C10B—C1	−179.6 (2)
N5—C4A—C10B—C10A	−2.3 (2)	C5A—C10A—C10B—C4A	1.5 (2)

C4—C4A—C10B—C1	−2.0 (3)	C10—C10A—C10B—C1	2.4 (4)
C4—C4A—C10B—C10A	177.16 (19)	C10—C10A—C10B—C4A	−176.58 (19)