Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(8-Chloro-6,9-dimethyl-2,3,4,9-tetrahydro-1*H*-carbazol-4-yl)acetaldehyde

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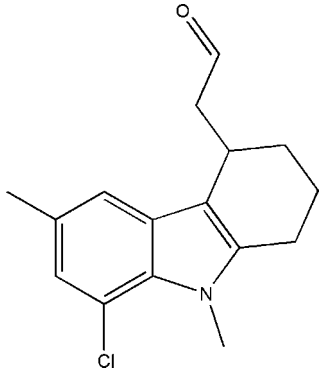
Received 14 November 2007; accepted 14 November 2007

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.057; wR factor = 0.169; data-to-parameter ratio = 14.1.

The geometric parameters of the title compound, $C_{16}H_{18}ClNO$, are in the usual ranges. The indole ring system is essentially planar and the attached six-membered saturated ring adopts a half-chair conformation. The crystal structure exhibits C—H$\cdots\pi$ interactions.

Related literature

For related literature, see: Allen *et al.* (1987); Liddell (2002).



Experimental

Crystal data

$C_{16}H_{18}ClNO$
M_r = 275.76
Triclinic, $P\overline{1}$
a = 8.0456 (5) Å
b = 9.5454 (4) Å
c = 9.5698 (4) Å
α = 90.094 (2)°
β = 104.405 (2)°
γ = 93.273 (2)°
V = 710.58 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 296 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
Absorption correction: none
3936 measured reflections
2452 independent reflections
1942 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.169
S = 1.09
2452 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C15—H15$B\cdots Cg1^{i}$	0.97	2.83	3.675	146

Symmetry code: (i) $-x+1, -y+1, -z+1$. $Cg1$ is the centroid of the C1–C6 ring.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

We are grateful to the National Science Foundation ofChina (20672040). We are also grateful to the Central China Normal University and Professor Xiao Wenjing.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2618).

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  doi:10.1107/S1600536807059156

supplementary materials

Acta Cryst. (2007). E**63**, o4818 [**doi:10.1107/S1600536807059156**]

2-(8-Chloro-6,9-dimethyl-2,3,4,9-tetrahydro-1*H*-carbazol-4-yl)acetaldehyde

Y.-Y. Lai, J.-R. Chen, G.-D. Jia and X. Tao

Comment

Polycyclic indoles are ubiquitous structural frameworks in the chemical literature, with various application identified throughout the pharmaceutical industry as well as among naturally occurring andbiological active molecules. As a result, considerable research effort has been directed toward their synthesis (Liddell, 2002). We report here the crystal structure of a polycyclic indole derivative, the title compound, (I).

In the crystal structure of (I), bond lengths and angles are within normal ranges (Allen *et al.* 1987)·The indole ring systems (N1/C1–C9) are close to planar, with a maximum deviation of 0.026 (2) A ° for atom C9.

Experimental

To a mixture of 5-Methyl-7-chloro-1-methyl-2-(4-pentenyl)-1 *H*-Indole(0.30 mmol), crotonaldehyde (1.5 mmol), and dichloroethane (3 ml), 2nd Hoveyda-Grubbs' catalyst (0.009 mmol) was added. The reaction mixture was then stirred underboiling dichloroethane. Upon the completion of reaction monitored by TLC, thesolvent was removed *in vacuo* to give pure cyclization product, 2-(8-Chloro-6,9-dimethyl-2,3,4,9-tetrahydro-1*H*-carbazol-4-yl) acetaldehyde, through column chromatography on silica gel(hexane/ ethyl acetate (10:1)). Crystals suitable for data collection were obtained by slow evaporation of an $CHCl_3$ solution at 283 K.

Refinement

All H atoms bonded to C atoms were initially located in difference Fourier maps and then constrained to their ideal geometry positions with C–H=0.96Å (methyl), 0.97Å (methylene), their U_{iso} values being set 1.5 times of U_{eq} (methyl C) and 1.2 times of U_{eq}(methylene).

Figures

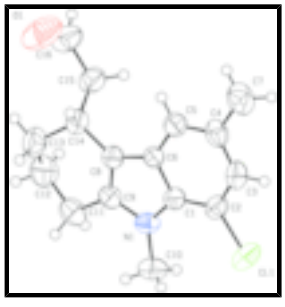

Fig. 1. View of a molecule of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by spheres of arbitrary radius.

2-(8-Chloro-6,9-dimethyl-2,3,4,9-tetrahydro-1 *H*-carbazol-4-yl)acetaldehyde

Crystal data

$C_{16}H_{18}ClNO$	$Z = 2$
$M_r = 275.76$	$F_{000} = 292$
Triclinic, $P\overline{1}$	$D_x = 1.289$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.0456$ (5) Å	Cell parameters from 1643 reflections
$b = 9.5454$ (4) Å	$\theta = 2.6$–24.8º
$c = 9.5698$ (4) Å	$\mu = 0.26$ mm^{-1}
$\alpha = 90.094$ (2)º	$T = 296$ (2) K
$\beta = 104.405$ (2)º	Block, colorless
$\gamma = 93.273$ (2)º	0.30 × 0.20 × 0.20 mm
$V = 710.58$ (6) Å^3	

Data collection

Bruker SMART 4K CCD area-detector diffractometer	1942 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.014$
Monochromator: graphite	$\theta_{max} = 25.0$º
$T = 296(2)$ K	$\theta_{min} = 2.2$º
φ and ω scans	$h = -9\rightarrow9$
Absorption correction: none	$k = -11\rightarrow11$
3936 measured reflections	$l = -7\rightarrow11$
2452 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.057$	H-atom parameters constrained
$wR(F^2) = 0.169$	$w = 1/[\sigma^2(F_o^2) + (0.0847P)^2 + 0.2869P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} = 0.011$
2452 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
174 parameters	$\Delta\rho_{min} = -0.20$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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}
C1	0.4323 (3)	0.8348 (2)	0.5724 (3)	0.0429 (6)
C2	0.4365 (3)	0.8845 (3)	0.7104 (3)	0.0488 (6)
C3	0.5824 (4)	0.8723 (3)	0.8196 (3)	0.0562 (7)
H3	0.5854	0.9077	0.9109	0.067*
C4	0.7269 (4)	0.8084 (3)	0.7981 (3)	0.0568 (7)
C5	0.7235 (3)	0.7596 (3)	0.6618 (3)	0.0514 (7)
H5	0.8189	0.7183	0.6452	0.062*
C6	0.5770 (3)	0.7718 (2)	0.5478 (3)	0.0402 (6)
C7	0.8822 (4)	0.7952 (5)	0.9222 (4)	0.0844 (11)
H7A	0.9567	0.7302	0.8963	0.127*
H7B	0.8462	0.7616	1.0052	0.127*
H7C	0.9427	0.8853	0.9443	0.127*
C8	0.5351 (3)	0.7281 (3)	0.4008 (3)	0.0441 (6)
C9	0.3708 (3)	0.7620 (3)	0.3424 (3)	0.0472 (6)
C10	0.1375 (4)	0.8860 (3)	0.4133 (4)	0.0667 (8)
H10A	0.0629	0.8300	0.4581	0.100*
H10B	0.0898	0.8858	0.3109	0.100*
H10C	0.1489	0.9805	0.4505	0.100*
C11	0.2720 (4)	0.7353 (3)	0.1905 (3)	0.0650 (8)
H11A	0.2615	0.8226	0.1381	0.078*
H11B	0.1574	0.6960	0.1881	0.078*
C12	0.3671 (5)	0.6324 (4)	0.1206 (4)	0.0847 (11)
H12A	0.3464	0.5385	0.1535	0.102*
H12B	0.3214	0.6329	0.0168	0.102*
C13	0.5577 (5)	0.6675 (4)	0.1554 (3)	0.0760 (10)
H13A	0.5783	0.7616	0.1227	0.091*
H13B	0.6100	0.6031	0.1029	0.091*
C14	0.6443 (4)	0.6595 (3)	0.3172 (3)	0.0520 (7)
H14	0.7543	0.7147	0.3351	0.062*
C15	0.6830 (4)	0.5107 (3)	0.3646 (4)	0.0635 (8)
H15A	0.6978	0.5067	0.4682	0.076*
H15B	0.5838	0.4491	0.3202	0.076*
C16	0.8367 (5)	0.4553 (4)	0.3295 (4)	0.0762 (10)

H16	0.8492	0.3594	0.3404	0.091*
Cl1	0.26043 (11)	0.95592 (9)	0.75368 (10)	0.0754 (3)
N1	0.3054 (3)	0.8278 (2)	0.4444 (2)	0.0493 (6)
O1	0.9436 (4)	0.5193 (3)	0.2898 (4)	0.1204 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0414 (13)	0.0410 (13)	0.0474 (14)	0.0000 (10)	0.0135 (11)	0.0031 (10)
C2	0.0532 (15)	0.0430 (14)	0.0545 (16)	0.0024 (11)	0.0218 (13)	−0.0009 (11)
C3	0.0683 (18)	0.0589 (17)	0.0428 (15)	−0.0061 (13)	0.0187 (13)	−0.0043 (12)
C4	0.0516 (16)	0.0705 (18)	0.0438 (15)	−0.0075 (13)	0.0062 (12)	0.0052 (13)
C5	0.0446 (14)	0.0587 (16)	0.0521 (16)	0.0027 (12)	0.0142 (12)	0.0051 (12)
C6	0.0374 (12)	0.0426 (13)	0.0404 (13)	0.0018 (10)	0.0096 (10)	0.0030 (10)
C7	0.068 (2)	0.123 (3)	0.0525 (19)	0.002 (2)	−0.0022 (16)	0.0024 (19)
C8	0.0453 (14)	0.0429 (13)	0.0444 (14)	0.0001 (10)	0.0123 (11)	0.0007 (10)
C9	0.0459 (15)	0.0465 (14)	0.0458 (14)	−0.0002 (11)	0.0059 (11)	0.0011 (11)
C10	0.0511 (17)	0.0658 (18)	0.081 (2)	0.0173 (14)	0.0094 (15)	0.0070 (16)
C11	0.0619 (18)	0.071 (2)	0.0518 (17)	−0.0004 (15)	−0.0035 (14)	0.0010 (14)
C12	0.098 (3)	0.094 (3)	0.0523 (19)	0.008 (2)	−0.0006 (18)	−0.0190 (17)
C13	0.102 (3)	0.078 (2)	0.0533 (18)	0.0097 (19)	0.0290 (18)	−0.0069 (16)
C14	0.0558 (16)	0.0516 (15)	0.0517 (16)	−0.0004 (12)	0.0204 (13)	−0.0016 (12)
C15	0.0661 (19)	0.0541 (17)	0.079 (2)	0.0069 (14)	0.0335 (16)	0.0052 (15)
C16	0.075 (2)	0.0592 (19)	0.103 (3)	0.0086 (16)	0.037 (2)	−0.0016 (18)
Cl1	0.0758 (6)	0.0752 (6)	0.0866 (6)	0.0158 (4)	0.0395 (5)	−0.0139 (4)
N1	0.0387 (11)	0.0521 (13)	0.0544 (13)	0.0084 (9)	0.0053 (10)	0.0012 (10)
O1	0.092 (2)	0.102 (2)	0.192 (3)	0.0083 (17)	0.083 (2)	0.004 (2)

Geometric parameters (Å, °)

C1—N1	1.385 (3)	C10—H10A	0.9600
C1—C2	1.394 (4)	C10—H10B	0.9600
C1—C6	1.411 (3)	C10—H10C	0.9600
C2—C3	1.374 (4)	C11—C12	1.528 (5)
C2—Cl1	1.745 (3)	C11—H11A	0.9700
C3—C4	1.403 (4)	C11—H11B	0.9700
C3—H3	0.9300	C12—C13	1.504 (5)
C4—C5	1.377 (4)	C12—H12A	0.9700
C4—C7	1.506 (4)	C12—H12B	0.9700
C5—C6	1.403 (4)	C13—C14	1.536 (4)
C5—H5	0.9300	C13—H13A	0.9700
C6—C8	1.419 (3)	C13—H13B	0.9700
C7—H7A	0.9600	C14—C15	1.517 (4)
C7—H7B	0.9600	C14—H14	0.9800
C7—H7C	0.9600	C15—C16	1.484 (4)
C8—C9	1.359 (4)	C15—H15A	0.9700
C8—C14	1.501 (4)	C15—H15B	0.9700
C9—N1	1.384 (3)	C16—O1	1.166 (4)
C9—C11	1.487 (4)	C16—H16	0.9300

C10—N1	1.452 (3)		
N1—C1—C2	132.7 (2)	C9—C11—C12	108.5 (2)
N1—C1—C6	107.8 (2)	C9—C11—H11A	110.0
C2—C1—C6	119.4 (2)	C12—C11—H11A	110.0
C3—C2—C1	119.3 (3)	C9—C11—H11B	110.0
C3—C2—Cl1	117.8 (2)	C12—C11—H11B	110.0
C1—C2—Cl1	122.8 (2)	H11A—C11—H11B	108.4
C2—C3—C4	122.2 (3)	C13—C12—C11	112.7 (3)
C2—C3—H3	118.9	C13—C12—H12A	109.1
C4—C3—H3	118.9	C11—C12—H12A	109.1
C5—C4—C3	118.6 (3)	C13—C12—H12B	109.1
C5—C4—C7	121.3 (3)	C11—C12—H12B	109.1
C3—C4—C7	120.1 (3)	H12A—C12—H12B	107.8
C4—C5—C6	120.4 (3)	C12—C13—C14	113.0 (3)
C4—C5—H5	119.8	C12—C13—H13A	109.0
C6—C5—H5	119.8	C14—C13—H13A	109.0
C5—C6—C1	120.0 (2)	C12—C13—H13B	109.0
C5—C6—C8	132.8 (2)	C14—C13—H13B	109.0
C1—C6—C8	107.2 (2)	H13A—C13—H13B	107.8
C4—C7—H7A	109.5	C8—C14—C15	112.9 (2)
C4—C7—H7B	109.5	C8—C14—C13	108.8 (2)
H7A—C7—H7B	109.5	C15—C14—C13	112.5 (3)
C4—C7—H7C	109.5	C8—C14—H14	107.5
H7A—C7—H7C	109.5	C15—C14—H14	107.5
H7B—C7—H7C	109.5	C13—C14—H14	107.5
C9—C8—C6	106.9 (2)	C16—C15—C14	115.8 (2)
C9—C8—C14	123.8 (2)	C16—C15—H15A	108.3
C6—C8—C14	129.3 (2)	C14—C15—H15A	108.3
C8—C9—N1	110.5 (2)	C16—C15—H15B	108.3
C8—C9—C11	126.0 (3)	C14—C15—H15B	108.3
N1—C9—C11	123.5 (2)	H15A—C15—H15B	107.4
N1—C10—H10A	109.5	O1—C16—C15	126.9 (3)
N1—C10—H10B	109.5	O1—C16—H16	116.6
H10A—C10—H10B	109.5	C15—C16—H16	116.6
N1—C10—H10C	109.5	C9—N1—C1	107.5 (2)
H10A—C10—H10C	109.5	C9—N1—C10	124.0 (2)
H10B—C10—H10C	109.5	C1—N1—C10	128.3 (2)
N1—C1—C2—C3	177.5 (3)	C14—C8—C9—C11	1.2 (4)
C6—C1—C2—C3	0.7 (4)	C8—C9—C11—C12	13.8 (4)
N1—C1—C2—Cl1	0.2 (4)	N1—C9—C11—C12	−167.0 (3)
C6—C1—C2—Cl1	−176.65 (18)	C9—C11—C12—C13	−44.2 (4)
C1—C2—C3—C4	−1.5 (4)	C11—C12—C13—C14	62.7 (4)
Cl1—C2—C3—C4	175.9 (2)	C9—C8—C14—C15	−112.3 (3)
C2—C3—C4—C5	1.7 (4)	C6—C8—C14—C15	68.7 (3)
C2—C3—C4—C7	−178.8 (3)	C9—C8—C14—C13	13.3 (3)
C3—C4—C5—C6	−1.1 (4)	C6—C8—C14—C13	−165.7 (3)
C7—C4—C5—C6	179.4 (3)	C12—C13—C14—C8	−43.8 (3)
C4—C5—C6—C1	0.3 (4)	C12—C13—C14—C15	82.1 (3)

C4—C5—C6—C8	−177.7 (3)	C8—C14—C15—C16	−158.1 (3)
N1—C1—C6—C5	−177.6 (2)	C13—C14—C15—C16	78.2 (4)
C2—C1—C6—C5	0.0 (3)	C14—C15—C16—O1	12.3 (6)
N1—C1—C6—C8	0.8 (3)	C8—C9—N1—C1	−0.6 (3)
C2—C1—C6—C8	178.4 (2)	C11—C9—N1—C1	−179.9 (2)
C5—C6—C8—C9	177.0 (3)	C8—C9—N1—C10	175.4 (2)
C1—C6—C8—C9	−1.2 (3)	C11—C9—N1—C10	−3.9 (4)
C5—C6—C8—C14	−3.9 (4)	C2—C1—N1—C9	−177.3 (3)
C1—C6—C8—C14	177.9 (2)	C6—C1—N1—C9	−0.2 (3)
C6—C8—C9—N1	1.1 (3)	C2—C1—N1—C10	7.0 (5)
C14—C8—C9—N1	−178.1 (2)	C6—C1—N1—C10	−175.9 (3)
C6—C8—C9—C11	−179.7 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15B···Cg1^{i}	0.97	2.83	3.675	146

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

Fig. 1