

P. G. Aravindan,^a
S. Selvanayagam,^a
M. Yogavel,^a D. Velmurugan,^{a*}
K. Ravikumar,^b N. Nagarajan^c
and P. T. Perumal^c

^aDepartment of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, ^bLaboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and ^cDepartment of Chemistry, Central Leather Research Institute, Adyar 600 020, India

Correspondence e-mail: d_velu@yahoo.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.048
wR factor = 0.130
Data-to-parameter ratio = 17.0

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

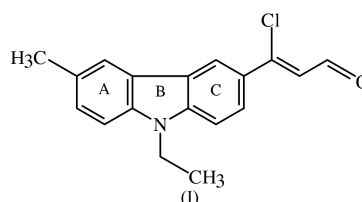
3-Chloro-3-(9-ethyl-6-methyl-9H-carbazol-3-yl)propenal

The carbazole moiety of the title molecule, $\text{C}_{18}\text{H}_{16}\text{ClNO}$, is planar to within $0.020 (2) \text{ \AA}$. The 3-chloropropenal substituent is coplanar with the carbazole ring system, whereas the *N*-ethyl substituent is inclined to it by $87.7 (2)^\circ$. The crystal packing is stabilized by weak $\pi-\pi$ interactions, $\text{C}-\text{H}\cdots\text{Cl}$ interactions and van der Waals forces.

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Comment

The bioactive carbazole ring system present in a number of natural products (Nakahara *et al.*, 2002) is responsible for their antimycobacterial, antifungal (Sunthitikawinsakul *et al.*, 2003), antiosteoporotic (Wang *et al.*, 2003), antitumoral (Martin *et al.*, 2002) and antioxidative (Tachibana *et al.*, 2001) activities. It has been found to have DNA-intercalating properties (Neidle, 1979; Aggarwal *et al.*, 1983). Carbazole behaves as a fluorescence carrier for the preparation of doxycycline sensors in pharmaceutical preparations. *N*-ethyl carbazole derivatives have been used for non-linear optical properties (Nesterov *et al.*, 2002). We report here the structure of the title compound, (I), a carbazole derivative.



The carbazole skeleton in (I) (Fig. 1) is planar to within $\pm 0.020 (2) \text{ \AA}$. The 3-chloropropenal substituent is coplanar with the carbazole ring system, with atoms C11, O1, C13, C14

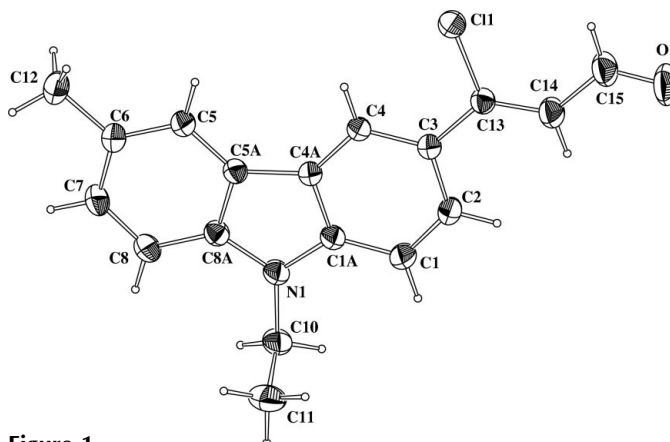


Figure 1
The structure of (I), showing 35% probability displacement ellipsoids and the atom-numbering scheme.

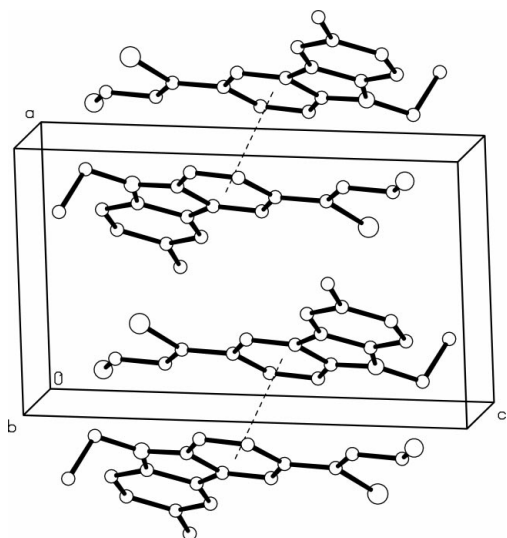


Figure 2
Packing of the molecules of (I), viewed down the *b* axis.

and C15 deviating from the carbazole plane by 0.022 (1), 0.014 (2), -0.011 (2), -0.046 (2) and -0.030 (2) Å, respectively. The C1A–N1–C10–C11 torsion angle of 87.7 (2)° shows how the *N*-ethyl substituent is oriented out of the carbazole ring system. The bond lengths and angles observed in (I) (Table 1) agree with those reported for related structures (Hökelek *et al.*, 2001*a,b*). A short contact between atoms H2 and H14 (2.04 Å) results in the widening of the C3–C13–C14 angle [126.5 (2)°] from the ideal value of 120°.

In addition to van der Waals forces and C–H...Cl interactions (Table 2), the crystal packing (Fig. 2) is stabilized by weak π – π interactions involving ring C. The molecules at (*x*, *y*, *z*) and ($-x$, $1 - y$, $1 - z$) are stacked with their C ring centroids separated by 3.751 (1) Å; the interplanar separation is 3.491 Å and the displacement is 1.88 Å (Glidewell *et al.*, 2002).

Experimental

To a stirred mixture of 3-methyl(9-ethyl-6-methylcarbazol) (0.5 g, 1 mmol) in dimethylformamide (7 ml) under ice-cold conditions, POCl₃ (3 mmol) was added and the mixture stirred at room temperature for 30 min. After completion of the reaction, the mixture was poured onto crushed ice, neutralized with sodium hydroxide solution (5%) and extracted with CHCl₃ (3 × 10 ml). The organic layer was separated, dried over anhydrous Na₂SO₄ and distilled under reduced pressure. The residue was then recrystallized from a mixture of ethyl acetate and petroleum ether (2:8) to give the title compound, (I).

Crystal data

C ₁₈ H ₁₆ ClNO	<i>Z</i> = 2
<i>M_r</i> = 297.77	<i>D_x</i> = 1.334 Mg m ⁻³
Triclinic, <i>P</i> $\bar{1}$	Mo <i>K</i> α radiation
<i>a</i> = 7.3191 (7) Å	Cell parameters from 1829 reflections
<i>b</i> = 8.9264 (9) Å	θ = 2.5–27.5°
<i>c</i> = 12.2024 (12) Å	μ = 0.26 mm ⁻¹
α = 105.247 (2)°	<i>T</i> = 293 (2) K
β = 91.771 (2)°	Block, yellow
γ = 104.378 (2)°	0.4 × 0.2 × 0.2 mm
<i>V</i> = 741.11 (13) Å ³	

Data collection

Siemens SMART CCD area-detector diffractometer
 ω scans
 4703 measured reflections
 3269 independent reflections
 2672 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.015$
 $\theta_{\text{max}} = 28^\circ$
 $h = -9 \rightarrow 9$
 $k = -11 \rightarrow 11$
 $l = -15 \rightarrow 11$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.130$
 $S = 1.02$
 3269 reflections
 192 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 0.1889P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.22 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

C11–C13	1.743 (2)	C13–C14	1.334 (3)
C3–C4	1.389 (2)	C1A–C1	1.390 (3)
C3–C2	1.412 (2)	C8A–C8	1.391 (3)
C3–C13	1.467 (2)	C14–C15	1.442 (3)
C4A–C4	1.389 (2)	C2–C1	1.371 (3)
C4A–C1A	1.412 (2)	C8–C7	1.375 (3)
C4A–C5A	1.445 (2)	C5–C6	1.384 (3)
N1–C1A	1.371 (2)	O1–C15	1.207 (3)
N1–C8A	1.384 (2)	C7–C6	1.403 (3)
N1–C10	1.456 (2)	C10–C11	1.504 (3)
C5A–C5	1.394 (2)	C6–C12	1.513 (3)
C5A–C8A	1.405 (2)		
C1A–N1–C8A	108.5 (1)	C14–C13–C3	126.5 (2)
C1A–N1–C10	126.1 (2)	C13–C14–C15	127.3 (2)
C8A–N1–C10	125.2 (2)		
C3–C13–C14–C15	-178.5 (2)	C8A–N1–C10–C11	-86.0 (2)
C1A–N1–C10–C11	87.7 (2)		

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C4–H4...Cl1	0.93	2.57	2.989 (2)	108
C15–H15...Cl1	0.93	2.68	3.048 (2)	105

H atoms were fixed geometrically and allowed to ride on their corresponding parent atoms, with C–H distances fixed in the range 0.93–0.97 Å and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{parent C})$ for the methyl H atoms and $1.2U_{\text{eq}}(\text{parent C})$ for the rest. A rotating group model was used for the methyl groups. Reflections were measured to θ_{max} of 27.97° with 92% completeness, but the data are 98% complete to 25°.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ZORTEP (Zsolnai, 1997) and PLATON (Spek, 1990); software used to prepare material for publication: SHELXL97 and PARST (Nardelli, 1995).

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