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3,6-Dibromo-9-ethyl-9*H*-carbazole

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.009 Å
R factor = 0.039
wR factor = 0.094
Data-to-parameter ratio = 11.6

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

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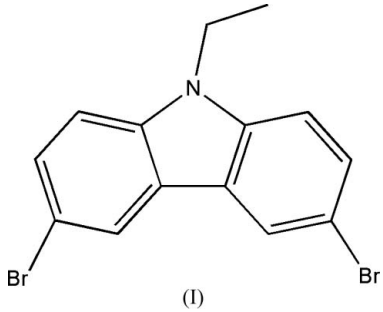
The title compound, $C_{14}H_{11}Br_2N$, was synthesized by *N*-alkylation of bromoethane with 3,6-dibromo-9*H*-carbazole. The carbazole ring system is essentially planar and makes a dihedral angle of 94.7 (3)° with the plane formed by the ethyl C and carbazole N atoms.

Comment

Carbazole derivatives substituted by *N*-alkylation are valuable for anticancer research (Buu-Hoï & Royer, 1950; Caulfield *et al.*, 2002; Morton *et al.*, 1983). In view of the increased toxicity to liver tissue developed in organic molecules by halogenation, emphasis has been placed on the synthesis of carbazole derivatives bearing halogen atoms (Buu-Hoï & Royer, 1951; Richard *et al.*, 2004). The title carbazole compound, (I), has recently been synthesized by *N*-alkylation of bromoethane with 3,6-dibromo-9*H*-carbazole. We present here its X-ray crystal structure.



(I)

The molecular structure of (I) is illustrated in Fig. 1. The carbazole ring system is essentially planar and the ethyl group is out of the carbazole plane. The dihedral angle between the carbazole plane and the plane formed by the ethyl C and carbazole N atoms is 94.7 (3)°.

Experimental

The title compound was prepared according to the procedure of Duan *et al.* (2005). 3,6-Dibromocarbazole (6.5 g, 20 mmol) was added to a dimethylformamide (DMF) solution (50 ml) of KOH (7.0 g) and the mixture was stirred for 40 min. A DMF solution (50 ml) of bromoethane (3.3 g, 30 mmol) was added dropwise with stirring. The resulting mixture was stirred at room temperature for 10 h and then poured into water (500 ml), yielding a white precipitate. The solid product was filtered, and washed with cold water. Crystallization from EtOH gave fine crystals of (I) (yield: 6.50 g, 92%; m.p. 412–414 K). ^{1}H NMR ($CDCl_3$): δ 1.33 (*t*, *J* = 7.1 Hz), 4.17 (*q*, *J* = 7.1 Hz, 2H), 7.16 (*d*, *J* = 8.5 Hz, 2H), 7.48 (*dd*, J_1 = 8.5 Hz, J_2 = 1.8 Hz, 2H), 8.13 (*d*, *J* = 1.8 Hz, 2H); ^{13}C NMR ($CDCl_3$): δ 13.64, 37.73, 110.05, 111.92, 123.20, 123.48, 128.95, 138.7. Recrystallization from a mixture

of chloroform (5 ml) and ethanol (5 ml) gave colorless single crystals of (I) after two weeks.

Crystal data

$C_{14}H_{11}Br_2N$
M_r = 353.06
Monoclinic, $P2_1$
a = 4.2588 (8) Å
b = 10.930 (2) Å
c = 13.724 (3) Å
β = 95.733 (3)°
V = 635.6 (2) Å^3
Z = 2
D_x = 1.845 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1746 reflections
θ = 3.0–25.7°
μ = 6.35 mm^{-1}
T = 294 (2) K
Block, colorless
0.28 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.176, T_{max} = 0.281
3520 measured reflections
1804 independent reflections
1470 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 26.3°
$h = -5 \rightarrow 5$
$k = -8 \rightarrow 13$
$l = -14 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.094
S = 1.04
1804 reflections
155 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2 + 0.263P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.66 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}
Absolute structure: Flack (1983), 444 Friedel Pairs
Flack parameter: 0.02 (2)

Table 1
Selected geometric parameters (Å).

Br1—C4	1.907 (7)	N1—C7	1.386 (8)
Br2—C10	1.908 (6)	N1—C13	1.453 (8)
N1—C1	1.371 (8)		

H atoms were positioned geometrically, with C—H = 0.93–0.97 Å and refined using a riding model [U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C)].

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

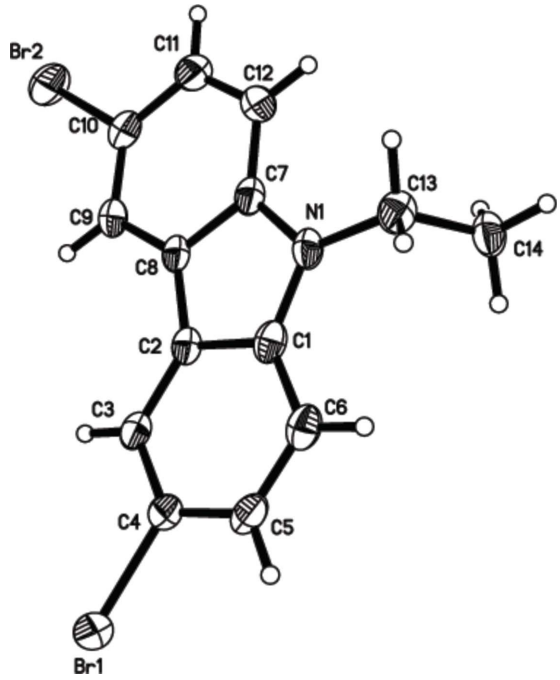


Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

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