Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,6-Dibromo-9-(4-chlorobenzyl)-9*H*-carbazole

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.035
wR factor = 0.098
Data-to-parameter ratio = 15.9

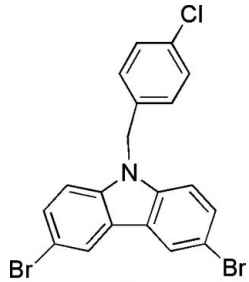
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 December 2005
Accepted 6 December 2005
Online 10 December 2005

The title compound, $C_{19}H_{12}Br_2ClN$, was synthesized by *N*-alkylation of 1-chloro-4-(chloromethyl)benzene with 3,6-dibromo-9*H*-carbazole. The carbazole ring system is essentially planar, with a mean deviation of 0.026 Å, and makes a dihedral angle of 75.16 (7)° with the plane of the benzene ring.

Comment

Carbazole derivatives substituted by *N*-alkylation show useful pharmaceutical properties (Buu-Hoï & Royer, 1950; Harfenist & Joyner, 1983; Caulfield *et al.*, 2002; Harper *et al.*, 2002). In this paper, the structure of 3,6-dibromo-9-(4-chlorobenzyl)-9*H*-carbazole, (I), is reported; this was synthesized by *N*-alkylation of 1-chloro-4-(chloromethyl)benzene with 3,6-dibromo-9*H*-carbazole.



(I)

The carbazole ring system in (I) is essentially planar, with a mean deviation of 0.026 Å. The dihedral angle formed between the carbazole ring system and the plane of the benzene ring is 75.16 (7)°. The C—Cl distance is 1.738 (4) Å and the C—Br distances are 1.906 (4) and 1.909 (3) Å.

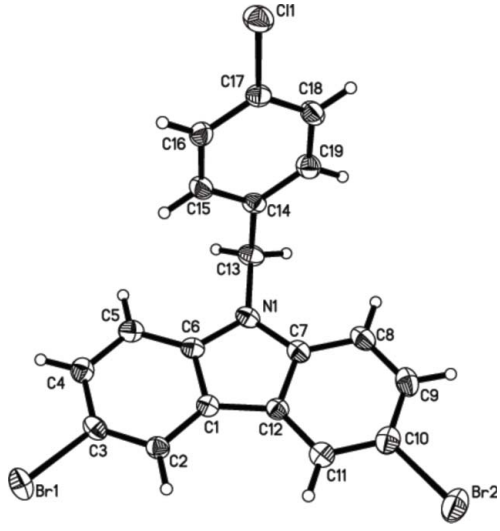


Figure 1
A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

Experimental

The title compound was prepared according to the procedure of Duan *et al.* (2005). A solution of potassium hydroxide (7.0 g) in dimethylformamide (50 ml) was stirred at room temperature for 20 min. 3,6-Dibromo-9*H*-carbazole (6.50 g, 20 mmol) (Smith *et al.*, 1992) was added and the mixture stirred for a further 40 min. A solution of 1-chloro-4-(chloromethyl)benzene (4.83 g, 30 mmol) in dimethylformamide (50 ml) was added dropwise with stirring. The resulting mixture was then stirred at room temperature for 12 h and poured into water (500 ml), yielding a white precipitate. The solid product was filtered off, washed with cold water and recrystallized from EtOH, giving crystals of (I) (yield: 7.88 g, 87.5%; m.p. 443–444 K). Compound (I) (40 mg) was dissolved in a mixture of chloroform (5 ml) and ethanol (5 ml) and the solution was kept at room temperature for 16 d. Natural evaporation of the solution gave colourless crystals suitable for X-ray analysis.

Crystal data

$C_{19}H_{12}Br_2ClN$
$M_r = 449.57$
Orthorhombic, *Pbca*
$a = 8.968$ (2) Å
$b = 16.690$ (3) Å
$c = 22.019$ (4) Å
$V = 3295.7$ (11) Å^3
$Z = 8$
$D_x = 1.812$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4904 reflections
$\theta = 2.6$–24.9°
$\mu = 5.08$ mm^{-1}
$T = 294$ (2) K
Block, colourless
0.28 × 0.26 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{min} = 0.269$, $T_{max} = 0.327$
17184 measured reflections
3315 independent reflections
2366 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 26.2^\circ$
$h = -10 \rightarrow 11$
$k = -8 \rightarrow 20$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.099$
$S = 1.07$
3315 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.6556P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}

All H atoms were included in the riding-model approximation, with C—H = 0.93 (aromatic) and 0.97 Å (methylene), and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*.

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