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

Xue-Min Duan,[a]* Peng-Mian Huang,[b] Jiang-Sheng Li,[b] Peng-Wu Zheng[a] and Xiao-Ji Wang[a]

[a]School of Pharmacy, Jiangxi Science & Technology Normal University, Nanchang 330013, People's Republic of China, and [b]College of Pharmaceuticals & Biotechnology, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: dxmlhp@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.038
wR factor = 0.114
Data-to-parameter ratio = 16.0

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

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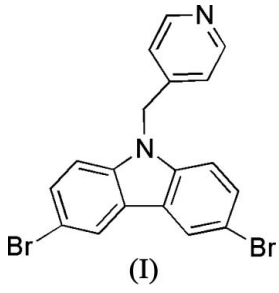
The title compound, $C_{18}H_{12}Br_2N_2$, was synthesized by *N*-alkylation of 4-chloromethylpyridine with 3,6-dibromo-9*H*-carbazole. The carbazole ring system is essentially planar, with a mean deviation of 0.012 Å, and makes a dihedral angle of 83.2 (8)° with the plane of the pyridine ring. In the crystal structure, weak C—H···Br interactions are observed.

Comment

Carbazole derivatives substituted by *N*-alkylation have useful pharmaceutical properties (Buu-Hoï & Royer, 1950; Harfenist & Joyner, 1983; Caulfield *et al.*, 2002; Harper *et al.*, 2002). In this paper we report the structure of 3,6-dibromo-9-(4-pyridylmethyl)-9*H*-carbazole, (I), which was synthesized by *N*-alkylation of 4-chloromethylpyridine with 3,6-dibromo-9*H*-carbazole.



(I)

The carbazole ring system in (I) is essentially planar, with a mean deviation of 0.012 Å, consistent with recent determinations of similar structures (Huang *et al.*, 2005; Duan, Huang *et al.*, 2005). The dihedral angle formed between the carbazole ring system and the plane of the pyridine ring is 83.2 (8)°. The C—Br distances are in the range 1.903 (3)–1.901 (4)%A, consistent with the literature (Allen *et al.*, 1987).

Experimental

The title compound was prepared according to the procedure of Duan, Han *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 4-chloromethylpyridine(3.83 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 dichlormethane and EtOH (1:1 *v*/*v*), giving crystals of (I). Yield: 7.29 g (87.6%); m.p. 510–511 K. Compound (I) (40 mg) was dissolved in a mixture of chloroform (5 ml) and ethanol (3 ml) and the solution

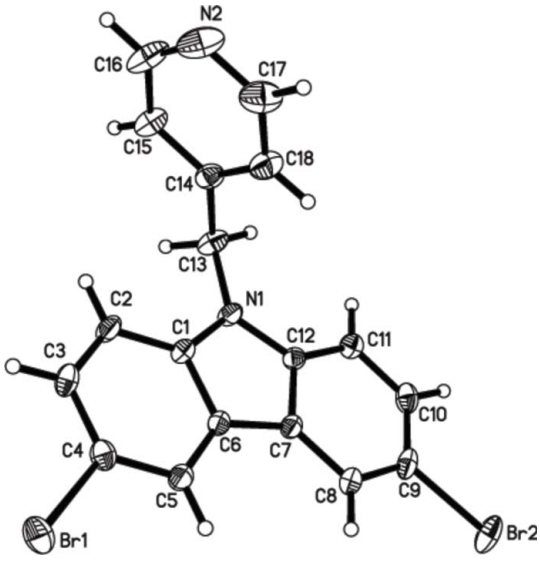


Figure 1
The molecular structure of (I), with displacement ellopsoids drawn at the 30% probability level.

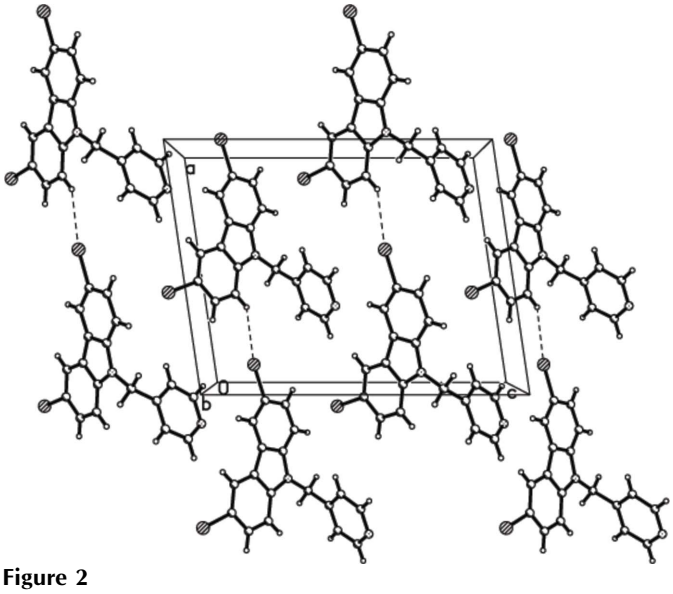


Figure 2
Packing of the title compound, viewed along [010]. Dashed lines indicate hydrogen bonds.

was kept at room temperature for 16 d. Natural evaporation of the solution gave colourless crystals suitable for X-ray analysis.

Crystal data

$C_{18}H_{12}Br_2N_2$
M_r = 416.12
Monoclinic, $P2_1/n$
a = 11.266 (4) Å
b = 9.729 (4) Å
c = 14.367 (5) Å
β = 98.341 (6)°
V = 1558.1 (10) Å^3
Z = 4
D_x = 1.774 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3336 reflections
θ = 2.5–26.3°
μ = 5.20 mm^{-1}
T = 294 (2) K
Rod, colourless
0.32 × 0.26 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.240, T_{max} = 0.353
8543 measured reflections
3207 independent reflections
2376 reflections with $I > 2\sigma(I)$
R_{int} = 0.068
θ_{max} = 26.5°
$h = -14 \rightarrow 9$
$k = -12 \rightarrow 10$
$l = -17 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.114
S = 1.00
3207 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0619P)^2 + 0.1399P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0067 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···Br2^{i}	0.93	2.87	3.653 (4)	142

Symmetry code: (i) $x-1, y, z$.

All H atoms were included using the riding model approximation, with C—H = 0.93 (aromatic) and 0.97 (methylene) Å, 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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