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Key indicators

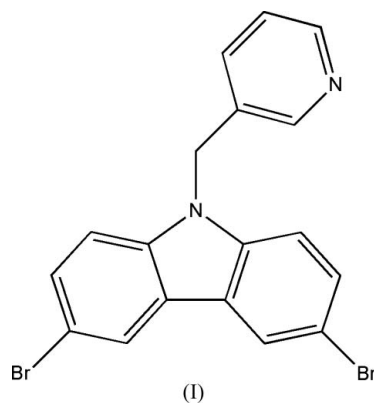
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.034
 wR factor = 0.078
Data-to-parameter ratio = 16.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

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

The title compound, $\text{C}_{18}\text{H}_{12}\text{Br}_2\text{N}_2$, was synthesized by *N*-alkylation of 3-(chloromethyl)pyridine with 3,6-dibromo-9H-carbazole. The carbazole ring system is essentially planar, with a mean deviation of 0.0138 Å, and forms a dihedral angle of 96.7 (8)° with the plane of the pyridine ring. In the crystal structure, π - π interactions and weak $\text{C}-\text{H}\cdots\text{Br}$ interactions are observed.

Comment

Carbazole derivatives substituted by *N*-alkylation possess valuable pharmaceutical properties (Buu-Hoï & Royer, 1950; Harfenist & Joyner, 1983; Caulfield *et al.*, 2002; Harper *et al.*, 2002). The title compound, 3,6-dibromo-9-(3-pyridylmethyl)-9H-carbazole, (I) (Fig. 1), was synthesized by *N*-alkylation of 3-(chloromethyl)pyridine with 3,6-dibromo-9H-carbazole.



The carbazole ring system is essentially planar, with a mean deviation of 0.0138 Å, consistent with previously reported values (Duan, Huang *et al.*, 2005). The dihedral angle formed between the carbazole ring system and the plane of the pyridine ring is 96.7 (8)°. $\text{C}-\text{Br}$ distances are in the range 1.905 (3) to 1.907 (3) Å and are consistent with literature values (Allen *et al.*, 1987). In the crystal structure, π - π interactions are observed; the shortest, 3.519 Å, is between the N1/C1-C12 and C7-C12 rings of molecules related by (1 - x , - y , 2 - z). In addition, there are $\text{C}-\text{H}\cdots\text{Br}$ interactions, as shown in Fig. 2 and detailed in Table 1.

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-Dibromocarbazole (6.5 g, 20 mmol), prepared according to Smith *et al.* (1992), was added and the mixture stirred for a further 40 min. A solution of 3-(chloromethyl)pyridine (3.825 g, 30 mmol) in

Received 2 November 2005

Accepted 5 December 2005

Online 10 December 2005

dimethylformamide (50 ml) was added dropwise with stirring. The resulting mixture was then stirred at room temperature for 10 h and poured into water (500 ml), yielding a white precipitate. The solid product (I) was collected by filtration, washed with cold water and recrystallized from EtOH (yield 7.11 g, 85.5%; m.p. 488 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 18 d. Slow evaporation of the solution yielded colourless crystals suitable for X-ray analysis.

Crystal data

$C_{18}H_{12}Br_2N_2$
 $M_r = 416.12$
 Monoclinic, $P2_1/c$
 $a = 10.469$ (3) Å
 $b = 16.405$ (5) Å
 $c = 9.866$ (3) Å
 $\beta = 112.761$ (4)°
 $V = 1562.6$ (8) Å³
 $Z = 4$

$D_x = 1.769$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2796 reflections
 $\theta = 2.5$ – 26.4 °
 $\mu = 5.19$ mm⁻¹
 $T = 294$ (2) K
 Block, colourless
 $0.26 \times 0.22 \times 0.14$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 1997)
 $T_{\min} = 0.258$, $T_{\max} = 0.484$
 8670 measured reflections

3194 independent reflections
 2238 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$
 $\theta_{\text{max}} = 26.5$ °
 $h = -12 \rightarrow 13$
 $k = -16 \rightarrow 20$
 $l = -10 \rightarrow 12$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.078$
 $S = 1.00$
 3194 reflections
 200 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0276P)^2 + 0.8633P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.48$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.52$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0233 (8)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C11-H11\cdots Br1^i$	0.93	2.89	3.595 (3)	134

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

All H atoms were placed in calculated positions and refined using a riding model, with C–H = 0.93 (aromatic) and 0.97 (methylene) Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

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.

We gratefully acknowledge financial support from the Foundation for Excellent Young Teachers of Jiangxi Science & Technology Normal University.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.

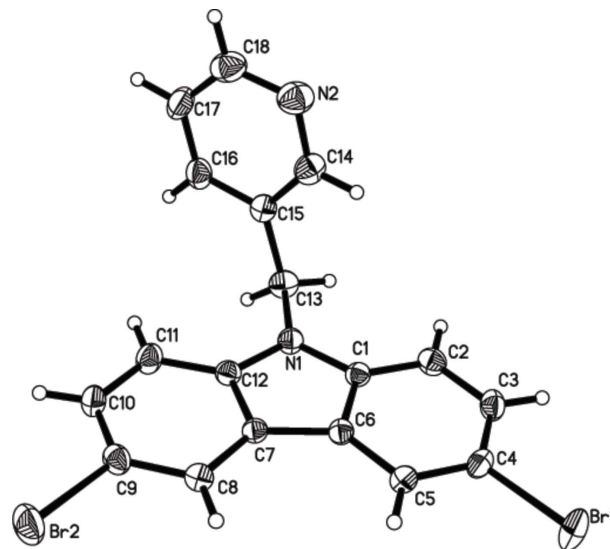


Figure 1
 The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

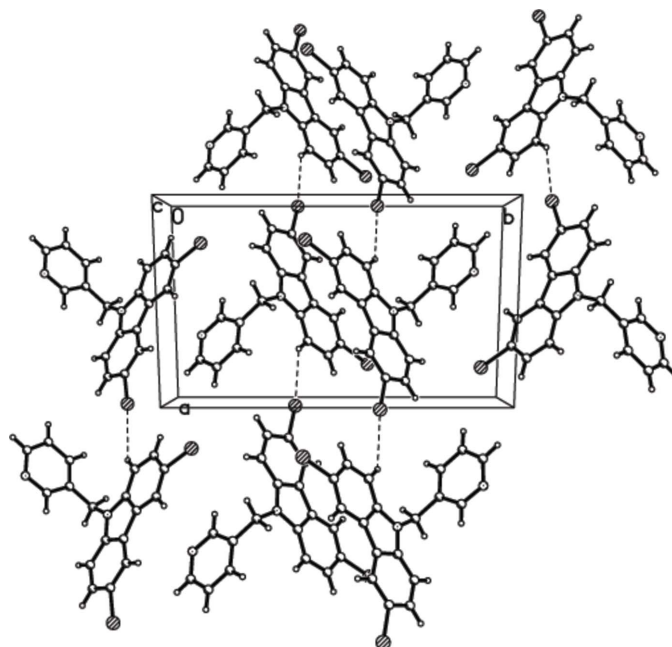


Figure 2
 Packing diagram of (I), viewed along [001]. Dashed lines indicate C–H...Br interactions.

- Bruker (1997). *SADABS, SMART, SAINT and SHELXTL*. Versions 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Buu-Hoi, N. P. & Royer, R. (1950). *J. Org. Chem.* **15**, 123–130.
- Caulfield, T., Cherrier, M. P., Combeau, C. & Mailliet, P. (2002). European Patent 1253141.
- Duan, X. M., Han, J., Chen, L. G., Xu, Y. J. & Li, Y. (2005). *Fine Chemicals*, **22**, 39–40, and 52.
- Duan, X. M., Huang, P. M., Zheng, P. W., Li, J. S. (2005). *Acta Cryst.* **E61**, o3361–o3363.
- Harfenist, M. & Joyner, C. T. (1983). US Patent 4379160.
- Harper, R. W., Lin, H. S. & Richett M. E. (2002). World Patent 02079154.
- Huang, P. M., Li, J. S., Duan, X. M., Zeng, T., Yan, X. L. (2005). *Acta Cryst.* **E61**, o2366–o2367.
- Sheldrick, G. M. (1997). *SHELXS97 and SHELXL97*. University of Göttingen, Germany.
- Smith, K., James, D. M., Mistry, A. G., Bye, M. R. & Faulkner, D. J. (1992). *Tetrahedron*, **48**, 7479–7488.