

9-Butyl-3-(2,6-diphenylpyridin-4-yl)-9H-carbazole

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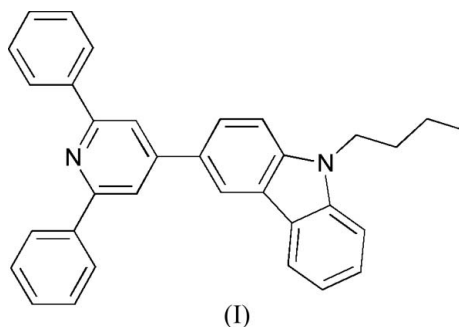
Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.060
 wR factor = 0.208
Data-to-parameter ratio = 13.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title molecule, $\text{C}_{33}\text{H}_{28}\text{N}_2$, the pyridine ring forms dihedral angles of 9.7 (1) and 13.0 (1)° with the two attached phenyl rings. The short distance of 3.585 (3) Å between the centroids of the pyridine rings of two neighbouring molecules reveals the existence of π - π interactions in the crystal structure.

Comment

Many materials including inorganic and organic crystals exhibit non-linear optical (NLO) properties (Cumpston *et al.*, 1999). Planar organic molecules with conjugative effects may demonstrate large NLO responses (Marder *et al.*, 1997). As part of our search for new organic materials with NLO properties, we have prepared the title compound, (I). We present here its crystal structure.



In (I) (Fig. 1), the bond lengths and angles show normal values (Allen *et al.*, 1987). The pyridine ring forms dihedral angles of 9.7 (1), 13.0 (1) and 33.4 (2)° with phenyl rings C28–C33 and C22–C27 and benzene ring C1–C6, respectively. The crystal packing (Fig. 2) shows a short $\text{Cg1} \cdots \text{Cg1}^i$ distance of 3.585 (3) Å [Cg1 is the centroid of the pyridine ring; symmetry code: (i) $1 - x, -y, 2 - z$], indicating the existence of π - π interactions in the crystal structure.

Experimental

For the preparation of 3-(9-butyl-9H-carbazol-6-yl)-1-phenylprop-2-en-1-one, a flask charged with a mixture of 9-butyl-3-carbaldehyde (12.6 g, 50 mmol), acetophenone (6.1 g, 50 mmol) and 2% aqueous sodium hydroxide (150 ml) was stirred vigorously at room temperature for 30 min, and was then heated at 333 K for 6 h. The reaction was monitored by thin-layer chromatography. When the reaction was complete, the mixture was cooled to room temperature. A light-yellow solid precipitated and was filtered off, washed thoroughly with water and air-dried to give 17.3 g (yield 98.0%) of the product. For the preparation of 9-butyl-3-(2,6-diphenylpyridin-4-yl)-9H-carbazole, acetophenone (1.8 g, 15 mmol), 3-(9-butyl-9H-carbazol-6-yl)-1-

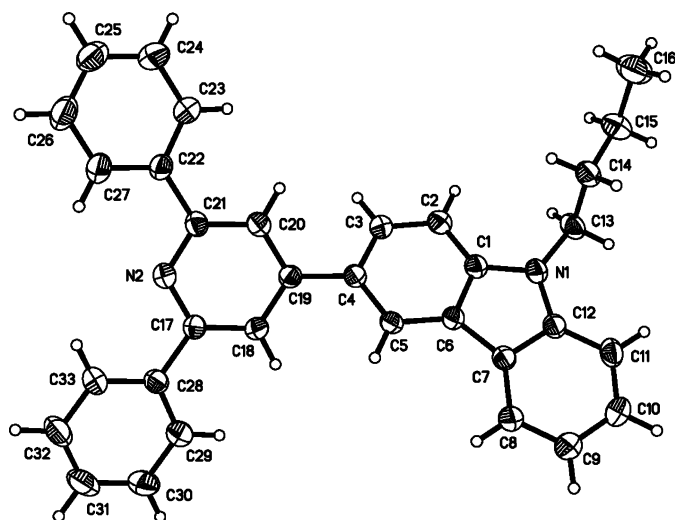


Figure 1
The molecular structure of (I), showing the atomic numbering and 30% probability displacement ellipsoids.

phenylprop-2-en-1-one (5.3 g, 15 mmol) and powdered NaOH (2.4 g, 60 mmol) were crushed together with a pestle and mortar for 2 h (Yang *et al.*, 2005). The resulting yellow powder was added to a stirred solution of ammonium acetate (10 g, excess) in ethanol (100 ml). The reaction mixture was heated at reflux for 10 h. Upon cooling to room temperature, a precipitate was filtered off, washed with water three times and dried to afford the product. Recrystallization from ethanol afforded pale-yellow block-shaped crystals. Single crystals suitable for X-ray diffraction were obtained by slow evaporation of a dichloromethane/2-propanol (2:1) solution. $^1\text{H NMR}$ (DMSO- d_6): 0.89 (*t*, 3H), 1.32 (*m*, 2H), 1.77 (*m*, 2H), 4.42 (*t*, 2H), 7.24 (*s*, 1H), 7.39 (*d*, 1H), 7.49 (overlap, 2H), 7.55 (*t*, 4H), 7.59 (*d*, 1H), 7.60 (*t*, 2H), 7.81 (*s*, 1H), 8.19 (*d*, 1H), 8.25 (*s*, 2H), 8.36 (*d*, 4H).

Crystal data

$\text{C}_{33}\text{H}_{28}\text{N}_2$
 $M_r = 452.57$
Monoclinic, $P2_1/c$
 $a = 11.489$ (3) Å
 $b = 15.221$ (4) Å
 $c = 15.099$ (4) Å
 $\beta = 111.104$ (4)°
 $V = 2463.1$ (11) Å³

$Z = 4$
 $D_x = 1.220$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.07$ mm⁻¹
 $T = 298$ (2) K
Block, pale yellow
 $0.45 \times 0.39 \times 0.36$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.969$, $T_{\max} = 0.975$

12777 measured reflections
4345 independent reflections
2082 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.073$
 $\theta_{\max} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.060$
 $wR(F^2) = 0.208$
 $S = 1.04$
4345 reflections
317 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0001P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.19$ e Å⁻³
 $\Delta\rho_{\min} = -0.18$ e Å⁻³
Extinction correction: SHELXL97
Extinction coefficient: 0.0023 (13)

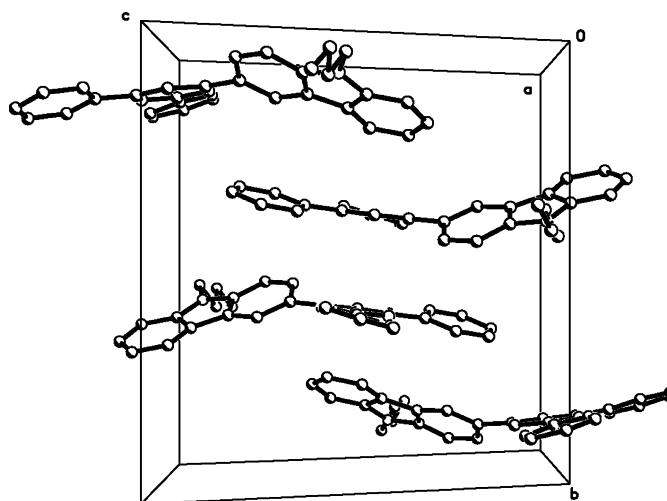


Figure 2
A packing diagram of (I), viewed along the a axis. H atoms have been omitted for clarity.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with $\text{C}-\text{H} = 0.93\text{--}0.97$ Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

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

This work was supported by the National Natural Science Foundation of China (grant Nos. 50532030 and 50325311), the Doctoral Program Foundation of the Ministry of Education of China, the Education Committee of Anhui Province (grant Nos. 2006 K J032A and 2006 K J135B), the Team for Scientific Innovation Foundation of Anhui Province (2006 K J007TD), and the Person with Ability Foundation of Anhui University. We also thank Professor D.-Q. Wang of Liao Cheng University for his assistance with the X-ray structure determination.

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