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Bis(9-ethylcarbazol-3-yl)ethane

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Key indicators

Single-crystal X-ray study

T = 295 K

Mean $\sigma(\text{C}-\text{C}) = 0.008 \text{ \AA}$

R factor = 0.056

wR factor = 0.132

Data-to-parameter ratio = 7.7

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

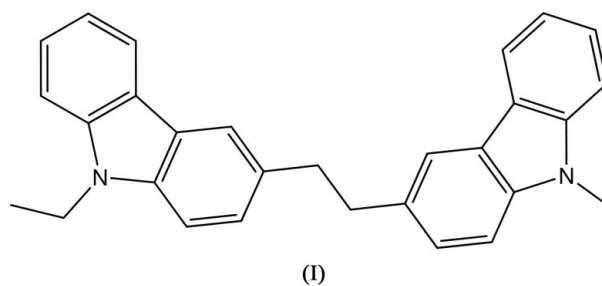
In the title compound, $\text{C}_{30}\text{H}_{28}\text{N}_2$, each carbazole skeleton is essentially planar. The planes of the two carbazole ring systems are nearly parallel, with a dihedral angle of $2.33 (19)^\circ$. The crystal packing is stabilized only by van der Waals forces and weak $\text{C}-\text{H} \cdots \pi$ interactions.

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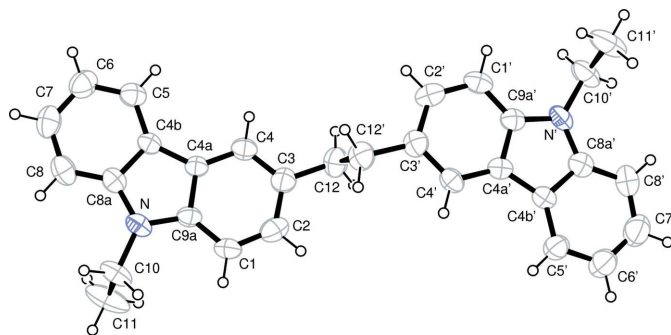
Comment

Polycarbazoles are among the most studied semiconducting polymers, due to their photoconduction properties (Loh *et al.*, 1991; Rocquin & Chevrot, 1997; Li *et al.*, 1998). The conductivities of these polymers are improved by the addition of certain dyes and electron acceptors as dopants (Iwatsuki & Arai, 1977; Hsiao *et al.*, 2001). Upon irradiation, interactions of electron donor-acceptor (EDA) groups along the polymer chain lead to the formation of charge-transfer (CT) complexes. Dicarbazolylalkanes serve as model compounds of related photoconducting polymers for investigating the nature of CT complexation of carbazoles with various electron acceptors, both in solution and in the solid state (Haderski *et al.*, 2000; Rocquin & Chevrot, 1997). We report here the crystal structure of the title compound, (I), which was synthesized to model poly-3-vinyl-*N*-ethylcarbazole in charge-transfer complexation studies.



The carbazole skeletons in (I) (Fig. 1) are essentially planar with only a slight distortion (r.m.s. deviations of the fitted atoms for both are 0.0436 \AA). The interplanar dihedral angle of $2.33 (19)^\circ$ indicates that the carbazolyl groups are almost parallel. The carbazolyl substituents show an *anti* conformation with respect to the $\text{C}12-\text{C}12'$ bond. The geometric parameters in (I) are unexceptional and agree with those of related dicarbazoles reported in the literature (Baker *et al.*, 1991; Asker & Masnovi, 2005). The torsion angles $\text{C}9\text{a}-\text{N}-\text{C}10-\text{C}11$ [$-86.0 (7)^\circ$] and $\text{C}9\text{a}'-\text{N}'-\text{C}10'-\text{C}11'$ [$84.0 (7)^\circ$] indicate that the *N*-ethyl substituents are almost perpendicular to the carbazole planes.

The crystal packing diagram (Fig. 2) shows that the structure is stabilized only by van der Waals forces and weak $\text{C}-$


Figure 1

The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 40% probability level.

H... π interactions, the strongest of which is C10—H10c...Cgⁱ (H10c...Cgⁱ = 2.73 Å), where Cg is the centroid of the pyrrole ring containing N [symmetry code: (i) $x - \frac{1}{2}, 1 - y, -z$]. No intermolecular π - π interactions are observed between the carbazole groups. The carbazole groups of two adjacent molecules are inclined toward each other, preventing such an interaction, possibly due to the orientations of the *N*-ethyl groups and the methylene chain.

Experimental

The synthesis of the title compound was accomplished *via* the catalytic hydrogenation of 1,2-bis(9-ethylcarbazol-3-yl)ethene, which was prepared according to a literature procedure, *via* TiCl₄/Zn-catalysed reductive coupling of 9-ethylcarbazole-3-carbaldehyde (Lynch *et al.*, 1997). In a 100 ml round-bottomed flask, a mixture prepared from 1.0 g (2.4 mmol) of 1,2-bis(9-ethylcarbazol-3-yl)ethene, 0.1 g of Pd-C (10%), and 30 ml of tetrahydrofuran was stirred under an H₂ atmosphere until the calculated amount of H₂ (55 ml) was consumed (about 2 h). The reaction mixture was then filtered and the solvent was evaporated. Column chromatography of the resulting solid, using alumina (80–200 mesh, activity III) as the carrier and dichloromethane/hexane (1:9 v/v) as eluent, yielded 0.96 g (95.57%) of (I) as colorless crystals (m.p. 454–455 K). ¹H NMR (300 MHz, CDCl₃): δ 8.05 (*d*, 7.86 Hz, 2H), 8.00 (*s*, 2H), 7.48–7.30 (*m*, 8H), 7.21 (*t*, 6.76 Hz, 2H), 4.35 (*q*, 7.31 Hz, 4H), 3.21 (*s*, 4H), 1.42 (*t*, 7.31 Hz, 6H).

Crystal data

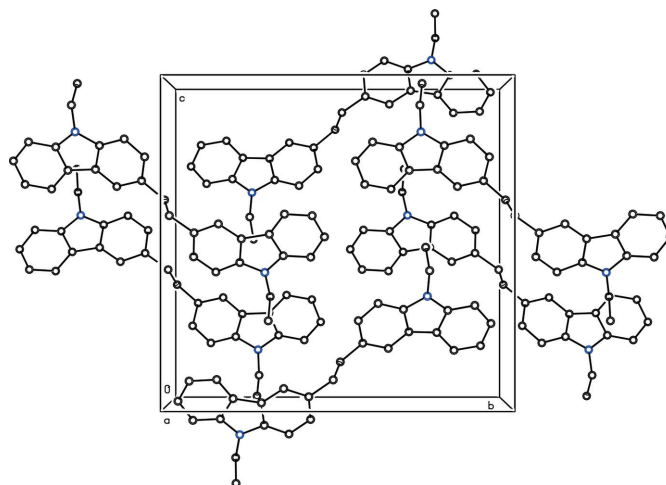
C₃₀H₂₈N₂
M_r = 416.54
 Orthorhombic, *P*₂₁*cn*
a = 8.1412 (5) Å
b = 17.2919 (19) Å
c = 16.434 (2) Å
V = 2313.5 (4) Å³
Z = 4
D_x = 1.196 Mg m⁻³

Mo *K* α radiation
 Cell parameters from 25 reflections
 θ = 1.7–25.1°
 μ = 0.07 mm⁻¹
T = 295 (2) K
 Cube, colorless
 0.50 × 0.50 × 0.50 mm

Data collection

Nonius CAD-4 diffractometer
 ω scans
 Absorption correction: none
 2214 measured reflections
 2214 independent reflections
 1136 reflections with *I* > 2 σ (*I*)
 θ_{\max} = 25.1°

h = 0 → 9
k = 0 → 20
l = 0 → 19
 3 standard reflections
 frequency: 120 min
 intensity decay: 0.7%


Figure 2

The molecular packing of (I), viewed down the *a* axis. H atoms have been omitted for clarity.

Refinement

Refinement on *F*²
R [*F*² > 2 σ (*F*²)] = 0.056
wR(*F*²) = 0.132
S = 0.92
 2214 reflections
 289 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0589P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.13 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.17 \text{ e \AA}^{-3}$

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93, 0.96 and 0.97 Å for aromatic, methyl and methylene H atoms, respectively, and with *U*_{iso}(H) = 1.5*U*_{eq}(C) for the methyl H atoms and 1.2*U*_{eq}(C) for the others.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC Software*; data reduction: *DATRD2* in *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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