

3-15-2006

9,9'-Diethyl-3,3'-di-9H-carbazolyl

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Recommended Citation

Asker, E., & Masnovi, J. (2006). 9,9'-diethyl-3,3'-di-9H-carbazolyl. *Acta Crystallographica Section E*, 62(3), o1133-o1135.
doi:10.1107/S160053680600571X

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9,9'-Diethyl-3,3'-di-9*H*-carbazolylErol Asker^{a*} and John Masnovi^b^aBalıkesir Üniversitesi, Necatibey Eğitim Fakültesi, 10100 Balıkesir, Turkey, and^bDepartment of Chemistry, Cleveland State University, Cleveland, OH 44115, USA

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Key indicators

Single-crystal X-ray study

 $T = 295\text{ K}$ Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$ R factor = 0.060 wR factor = 0.135

Data-to-parameter ratio = 13.6

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

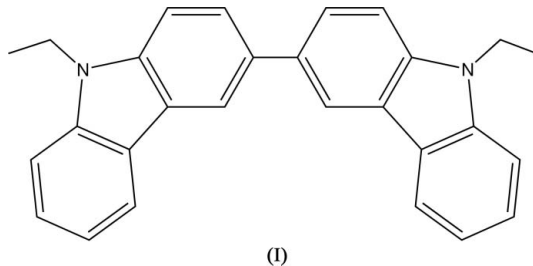
In the title compound, $\text{C}_{28}\text{H}_{24}\text{N}_2$, the carbazole ring systems are essentially planar to within 0.076 (3) Å. The dihedral angle between the planes of the ring systems is 40.38 (4)°. The contribution of intermolecular π - π interactions to the molecular stacking is observed.

Received 2 February 2006

Accepted 16 February 2006

Comment

Dicarbazolylalkanes, as the dimeric model compounds of poly-*N*-vinylcarbazole (PVK) and poly-3-vinylcarbazole (P3VK), have attracted some interest in studies dealing with photophysical properties of the corresponding polymers (Schildcrout *et al.*, 1991; Haderski *et al.*, 2000; Tani *et al.*, 2001). Crystal structures of some of the dicarbazolyl model compounds have already been reported (Baker *et al.*, 1991; Asker & Masnovi, 2005). In this paper, we report the structure of 9,9'-diethyl-3,3'-dicarbazolyl, (I), which was synthesized according to a literature procedure *via* oxidation of 9-ethylcarbazole by ferric chloride (Sadaki *et al.*, 1995).



The 13 atoms of each carbazole ring in (I) (Fig. 1) are essentially coplanar to within 0.076 (3) Å. Bond distances and angles in the carbazole rings (Table I) are in agreement with each other, as well as with those of related compounds reported in the literature (Baker *et al.*, 1991; Asker & Masnovi, 2005). The torsion angles $\text{C}9a-\text{N}-\text{C}10-\text{C}11$ [93.1 (4)°] and $\text{C}9a'-\text{N}'-\text{C}10'-\text{C}11'$ [82.3 (4)°] show how the *N*-ethyl substituents are oriented out of the carbazole ring system planes. Examination of the packing (Fig. 2) reveals the existence of π - π stacking interactions in the structure of (I), where the two carbazole groups of one molecule associate centrosymmetrically with one carbazole group of each of two adjacent molecules in such an orientation that their dipoles and ethyl groups point in opposite directions.

Experimental

The title compound, (I), was prepared according to the literature procedure *via* oxidation of 9-ethylcarbazole by ferric chloride (Sadaki *et al.*, 1995). To a solution of 9-ethylcarbazole (5.0 g, 0.026

mole) in dichloromethane (60 ml) in an oven-dried three-necked 250 ml flask, FeCl₃ (5.0 g, 0.031 mol) was added portionwise, with stirring, in an ice bath. The mixture was stirred for an additional hour at room temperature, during which time the solution became dark green. After 1 h, the reaction medium was carefully neutralized by dropwise addition of aqueous NaOH solution. After extraction of the mixture with additional dichloromethane (50 ml) and washing three times with water, the solvent was removed and the resulting solid was air-dried. Column chromatography of the crude product over basic alumina (80–200 mesh, activity III), using dichloromethane/hexane as eluant, yielded 2.2 g (43.7%) of colorless crystals [m.p. 464–465 K; literature 466–467 K (Chen *et al.*, 2000)]. ¹H NMR (300 MHz, CDCl₃): δ 8.44 (*d*, 1.64 Hz, 2H), 8.22 (*d*, 7.86 Hz, 2H), 7.86 (*d of d*, 8.59 and 1.83 Hz, 2H), 7.55–7.42 (*m*, 6H), 7.28 (*t*, 6.76 Hz, 2H), 4.43 (*q*, 7.31 Hz, 4H), 1.49 (*t*, 7.31 Hz, 6 H).

Crystal data

C₂₈H₂₄N₂
M_r = 388.49
 Tetragonal, *I*4₁/*a*
a = 22.6201 (8) Å
c = 16.3918 (12) Å
V = 8387.2 (7) Å³
Z = 16
D_x = 1.231 Mg m⁻³

Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 5.7–18.4°
 μ = 0.07 mm⁻¹
T = 295 (2) K
 Prism, colorless
 0.51 × 0.43 × 0.42 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω scans
 Absorption correction: none
 3689 measured reflections
 3689 independent reflections
 1481 reflections with *I* > 2σ(*I*)

θ_{\max} = 25.0°
h = 0 → 26
k = 0 → 26
l = 0 → 19
 3 standard reflections
 frequency: 120 min
 intensity decay: 1.3%

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.060
wR(*F*²) = 0.135
S = 0.86
 3689 reflections
 271 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} = 0.004
 Δρ_{max} = 0.15 e Å⁻³
 Δρ_{min} = -0.14 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

| | | | |
|-------------------|-----------|---------------|-----------|
| C4a–C4b | 1.444 (4) | C1'–C2' | 1.378 (4) |
| C3–C2 | 1.400 (4) | C1–C2 | 1.378 (4) |
| C3–C3' | 1.485 (4) | C4a'–C4b' | 1.446 (4) |
| C3'–C2' | 1.399 (4) | | |
| C4–C3–C2 | 118.0 (3) | C1–C9a–C4a | 122.1 (3) |
| N'–C9a'–C4a' | 109.4 (3) | N–C9a–C4a | 109.2 (3) |
| C1'–C9a'–C4a' | 120.6 (3) | C2–C1–C9a | 117.4 (4) |
| C4'–C3'–C2' | 117.2 (3) | C1–C2–C3 | 123.0 (4) |
| N'–C8a'–C4b' | 108.7 (3) | C1'–C2'–C3' | 123.4 (3) |
| N–C8a–C4b | 108.7 (3) | C7–C6–C5 | 120.9 (3) |
| C2'–C1'–C9a' | 117.8 (3) | C7'–C6'–C5' | 120.3 (4) |
| C9a'–N'–C10'–C11' | 82.3 (4) | C9a–N–C10–C11 | 93.1 (4) |

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

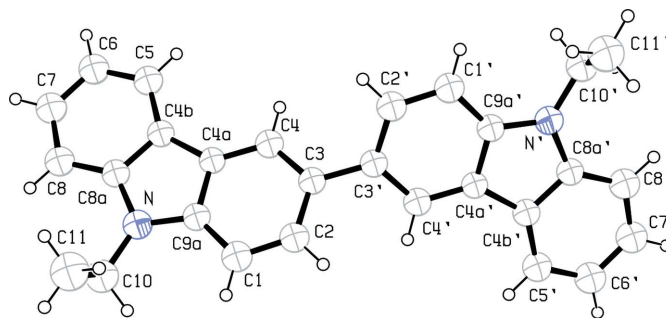


Figure 1 ORTEP-3 drawing (Farrugia, 1997) of (I) with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 40% probability level.

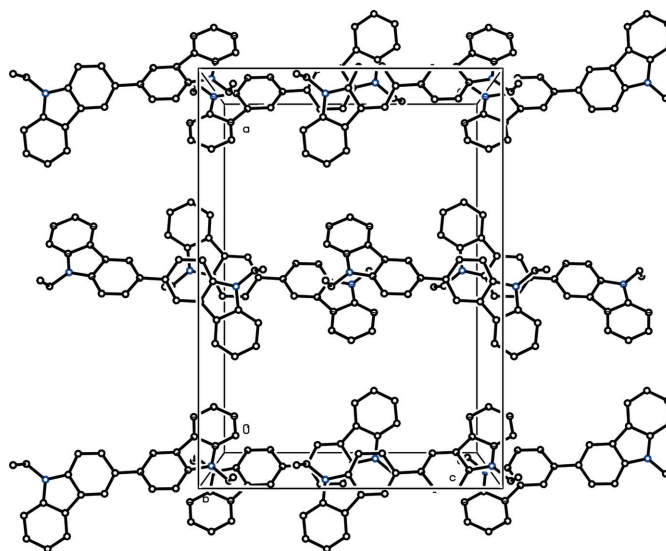


Figure 2 The two-dimensional layer structure of (I), viewed down the *b* axis. H atoms have been omitted for clarity.

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).

The authors thank the Turkish Ministry of Education and the CSU College of Graduate Studies for their support of this work.

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