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1,2-Bis(carbazol-9-ylmethylene)-3,4-diphenylcyclobutane

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.127
Data-to-parameter ratio = 13.3

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

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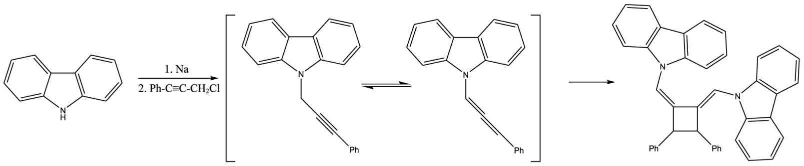
Carbazolyl fragments of the title compound, $C_{42}H_{30}N_2$, are involved in intramolecular stacking interactions between each other and intermolecular C—H$\cdots\pi$ hydrogen bonds. This probably causes some of their non-planarity.

Comment

We have previously demonstrated that the dimerization product of carbozolylallene is 1,2-bis(carbazol-9-ylmethylene)cylobutane (Kharanenko *et al.*, 2004). It can be generated both from propynylcarbazole and its precursor 1-(carbazolyl)-2,3-dichlorpropane. We have also observed the formation of this compound by alkylation of carbazole by propargyl bromide. Similarly, when 3-phenyl-2-propylchloride was alkylated, we obtained the product, (I), which IR and NMR spectroscopy confirmed was not an acetylene derivative. We now report the X-ray diffraction study of this compound.



(I)

The structure analysis reveals that the product of the reaction is 1,2-bis(carbazol-9-ylmethylene)-3,4-diphenylcylobutane. In the crystal structure, the title molecule has geometrical parameters which are very close to C_2 point-group symmetry. The approximate twofold axis intercepts the C29—C30 and C27—C28 bonds. The cyclobutane ring adopts a slightly twisted conformation, the C27—C30—C29—C28 torsion angle being 8.6 (2)°. For this reason, the C25═C27 and C26═C28 double bonds are not coplanar [C25—C27—C28—C26 = −16.1 (6)°]. The phenyl substituents have a *trans*-orientation with respect to the ring [C31—C30—C29—C37 = 101.3 (2)°].

The strong steric repulsion between the bulky substitutients at atoms C27 and C28 leads to significant deformation of the molecular geometry. The tricyclic fragments are not planar. The angles between the mean planes of the terminal phenyl rings are 5.8 (1) (C1$\cdots$C6—C7$\cdots$C12) and 3.4 (2)° (C13$\cdots$C18—C19$\cdots$C24). Rotation of the heterocyclic systems with respect to the C═C double bonds [C13—N2—C26—C28 and C7—N1—C25—C27 = −51.5 (4) and −49.3 (4)°, respectively] results in elongation of the C25—N1 and C26—N5 bonds (Table 1) compared with the mean value of 1.355 Å for Csp^2—N bonds (Bürgi & Dunitz, 1994). It is also observed that the exocyclic bond angles C25—C27—C28

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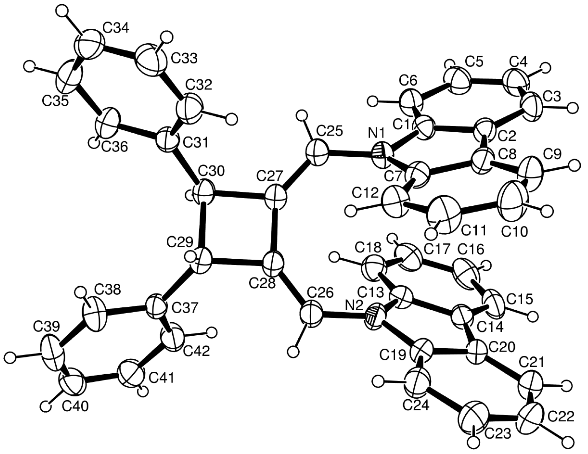

Figure 1
View of the title compound. The non-H atoms are shown with displacement ellipsoids drawn at the 30% probability level.

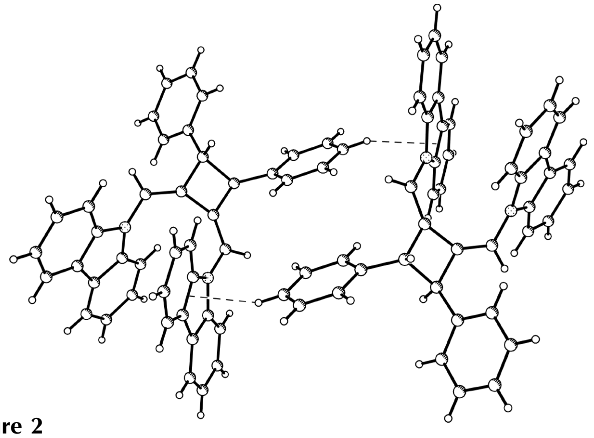

Figure 2
Centrosymmetric dimer of the title compound, linked by C—H$\cdots\pi$ hydrogen bonds.

and C27—C28—C26 are larger than the angles C25—C27—C30 and C26—C28—C29. The N atoms adopt a trigonal–pyramidal configuration; the sums of the bond angles at these atoms are 359.0 and 359.1° for N1 and N2, respectively. The slight distortions are probably due to stacking interactions between the carbazolyl fragments [the distance between the N atom and the mean plane of the opposite tricyclic ring system is ~ 2.98 Å and the angle between the planes is 25.50 (5)°].

In the crystal structure, molecules are linked into centrosymmetric dimers by C34—H34$\cdots\pi$ intermolecular hydrogen bonds (Fig. 2). Atom H34 is situated approximately above the mid-point of the C7$^{\rm i}$—C8$^{\rm i}$ [symmetry code: (i) $1 - x$, $1 - y$, $1 - z$] bond (designated X). The hydrogen-bond parameters are H34$\cdots X$ 2.74 Å and C34—H34$\cdots X$ 156°.

Experimental

The title compound was prepared according to the procedure of Kharanenko *et al.* (2004). Suitable crystals were obtained by evaporation of a pyridine solution (m.p. 391–392 K). Spectroscopic analysis, IR (KBr, ν, sm^{-1}): 3040, 1930, 1890, 1780, 1640, 1600, 1490, 1450, 1330, 1230, 740, 720; ^{1}H NMR (py-d_5, p.p.m.): 4.85 (2H, *s*, CH), 6.95 (2H, *s*, CH), 7.1–7.8 (26H, *m*, H-aromatic); analysis, calculated for $C_{42}H_{30}N_2$: C 89.5, H 5.4, N 5.1%; found: C 89.68, H 5.34, N 4.98%.

Crystal data

$C_{42}H_{30}N_2$
$M_r = 562.68$
Triclinic, $P\overline{1}$
$a = 8.830$ (2) Å
$b = 11.932$ (2) Å
$c = 14.888$ (3) Å
$\alpha = 90.94$ (2)°
$\beta = 105.58$ (2)°
$\gamma = 92.12$ (2)°
$V = 1509.4$ (5) Å^3

$Z = 2$
$D_x = 1.238$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 24 reflections
$\theta = 10$–12°
$\mu = 0.07$ mm^{-1}
$T = 293$ (2) K
Needle, colourless
0.40 × 0.20 × 0.20 mm

Data collection

Siemens *P*3/*PC* diffractometer
θ–2θ scans
Absorption correction: none
5672 measured reflections
5298 independent reflections
2469 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.044$

$\theta_{\rm max} = 25.1°$
$h = 0 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 17$
2 standard reflections every 98 reflections intensity decay: 5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.128$
$S = 0.96$
5298 reflections
397 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.12$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C25	1.403 (3)	C27—C28	1.463 (3)
N2—C26	1.400 (3)	C27—C30	1.530 (3)
C25—C27	1.333 (3)	C28—C29	1.528 (3)
C26—C28	1.332 (3)	C29—C30	1.585 (3)
C7—N1—C25	127.7 (2)	C25—C27—C28	139.9 (2)
C7—N1—C1	107.61 (18)	C25—C27—C30	127.8 (2)
C25—N1—C1	123.7 (2)	C28—C27—C30	92.11 (18)
C19—N2—C26	123.1 (2)	C26—C28—C27	139.7 (2)
C19—N2—C13	107.95 (19)	C26—C28—C29	128.5 (2)
C26—N2—C13	128.1 (2)	C27—C28—C29	91.72 (17)
C27—C25—N1	127.1 (2)	C28—C29—C30	87.61 (17)
C28—C26—N2	127.5 (2)	C27—C30—C29	87.16 (16)

All H atoms were placed in calculated positions (C—H = 0.93–0.98 Å) and included in the refinement in the riding-model approximation, with $U_{\rm iso} = 1.2$ times $U_{\rm eq}$ of the carrier atom.

Data collection: *P*3 (Siemens, 1989); cell refinement: *P*3; data reduction: *XDISK* (Siemens, 1991); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1991); software used to prepare material for publication: *SHELXL*97.

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