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Key indicators

Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.072
 wR factor = 0.161
Data-to-parameter ratio = 16.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**1,9-Diazaoctacyclo[22.6.1.1^{3,7}.1^{9,16}.2^{18,31}.0^{10,15}.-2^{21,16}.0^{24,29}]tetratriaconta-3,5,7(30),10(15),11,-13,16(31),17,19,21,23(36),24(29),25,27,32,34-hexadecaene**

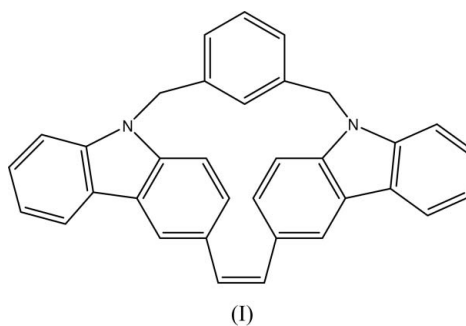
In the title compound, $\text{C}_{34}\text{H}_{24}\text{N}_2$, both carbazole ring systems are essentially planar and their planes form a dihedral angle of 31.70 (5)°. The benzene ring forms dihedral angles of 88.31 (5) and 79.58 (5)° with the carbazole ring systems. A π - π interaction [3.8062 (15) Å] is observed in the molecular structure, but no significant intermolecular interactions are found in the crystal structure.

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Comment

Functionalized carbazoles are of considerable interest as bioactive compounds that display a broad spectrum of pharmacological properties (Knolker & Reddy, 2002). The carbazole system is a highly photoactive nucleus which has potential applications in xerography and electro-imaging (Pearson & Stolka, 1981). The synthesis and charge transfer complexation behavior of carbazole-based cyclophanes such as [3,3][3,9]-carbazolophanes (Tani *et al.*, 2001), [2,2]paracyclo(3,6)-carbazolophanes (Tani *et al.*, 2002) and *N*-aryl carbazolophanes (Nakamura *et al.*, 2004) have been reported. Host-guest complexation behavior of the carbazolopyridinophane with N_2H_4 has also been reported (Brown *et al.*, 2005). The design and synthesis of highly rigid cavities and their exciplex formations of carbazole donor and some acceptor groups such as terephthalate (Tani *et al.*, 2003) and dicyanobenzene (Kanada *et al.*, 2000) derivatives are also known. We report here the crystal structure of the title compound, (I).



The non-H atoms of each carbazole ring in (I) (Fig. 1) are essentially coplanar, with maximum deviations of 0.078 (2) Å for atom C9 in the N1/C1–C12 ring system, and 0.107 (2) Å for atom C15 in the N2/C15–C26 ring system. Bond distances and angles in the carbazole units (Table 1) are in agreement with each other, as well as with those reported for carbazole derivatives (Duan *et al.*, 2006; Cui *et al.*, 2006). The only notable distinction is that the C3–C4 [1.385 (5) Å] bond is shorter

than the corresponding C23–C24 [1.406 (4) Å] bond, and the C4–C5 [1.398 (5) Å] bond is longer than the corresponding C22–C23 [1.377 (4) Å] bond. The dihedral angle formed between the planes of the two carbazole fragments is 31.70 (5)°. The dihedral angles formed by the N1/C1–C12 and N2/C15–C26 carbazole planes with the C28–C33 benzene ring are 88.31 (5) and 79.58 (5)°, respectively. In the molecular structure, a π – π interaction is formed between the C7–C12 and C15–C20 rings, with centroids separated by 3.8062 (15) Å.

No significant intermolecular π – π interactions involving the aromatic rings or hydrogen-bonding interactions are found in the crystal structure.

Experimental

TiCl₄ (19.5 mmol), Zn (39.0 mmol) and a few drops of pyridine were added to tetrahydrofuran (THF, 200 ml). The mixture was refluxed for 45 min and 9-[3-(3-formyl-9*H*-carbazole-9-yl)methyl]-3-formyl-9*H*-carbazole (0.98 mmol) was added. The reaction mixture was refluxed overnight, cooled, quenched with K₂CO₃ and filtered, and the THF was removed *in vacuo*. The residue was extracted with chloroform (3 × 100 ml), washed with water (2 × 100 ml) and brine (150 ml), and dried over anhydrous Na₂SO₄. The solvent was removed *in vacuo* and the residue was subjected to column chromatography (SiO₂) using hexane and chloroform (4:1). The compound was recrystallized from chloroform by slow evaporation.

Crystal data

C ₃₄ H ₂₄ N ₂	Z = 4
<i>M_r</i> = 460.55	<i>D_x</i> = 1.313 Mg m ^{−3}
Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Mo <i>K</i> α radiation
<i>a</i> = 20.1137 (10) Å	μ = 0.08 mm ^{−1}
<i>b</i> = 11.5719 (6) Å	<i>T</i> = 100 (2) K
<i>c</i> = 10.0137 (5) Å	Plate, colorless
β = 91.350 (3)°	0.31 × 0.16 × 0.07 mm
<i>V</i> = 2330.1 (2) Å ³	

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	29997 measured reflections
φ and ω scans	5328 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 2005)	3241 reflections with <i>I</i> > 2σ(<i>I</i>)
<i>T</i> _{min} = 0.977, <i>T</i> _{max} = 0.995	<i>R</i> _{int} = 0.111
	θ_{\max} = 27.5°

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2 + 1.152P]$
$R[F^2 > 2\sigma(F^2)] = 0.072$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.161$	(Δ/σ) _{max} = 0.001
<i>S</i> = 1.06	$\Delta\rho_{\max} = 0.28 \text{ e } \text{Å}^{-3}$
5328 reflections	$\Delta\rho_{\min} = -0.28 \text{ e } \text{Å}^{-3}$
325 parameters	
H-atom parameters constrained	

H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C–H distances of 0.95 (aromatic) and 0.99 Å (methylene). The *U*_{iso}(H) values were constrained to be 1.2*U*_{eq} of the carrier atoms.

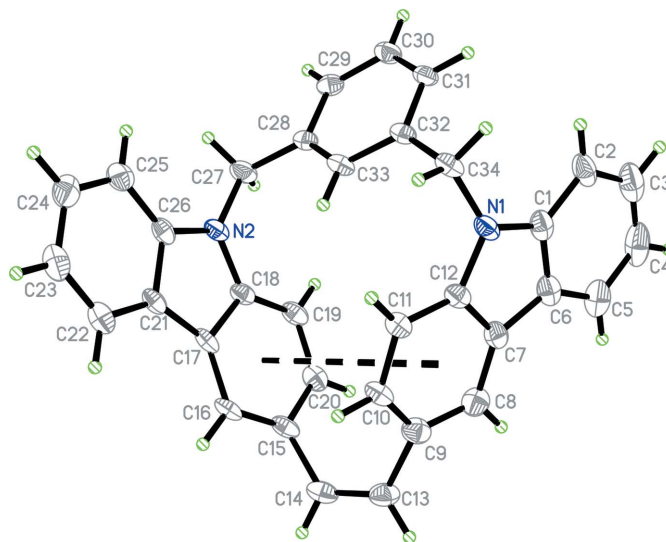


Figure 1

The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The π – π interaction is indicated by a dashed line.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 1998); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2003).

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