

9-(2,5-Dimethoxybenzyl)-9H-carbazole: sheets built from C—H... π and π — π interactionsG. Senthil Kumar,^a
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Key indicators

Single-crystal X-ray study

T = 100 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.046

wR factor = 0.137

Data-to-parameter ratio = 22.5

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

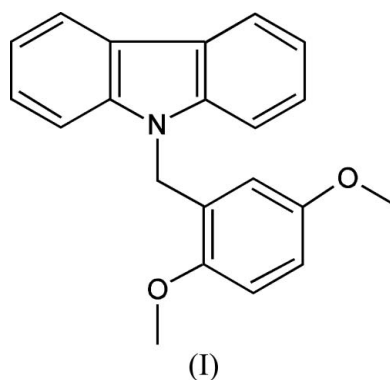
In the title compound, $\text{C}_{21}\text{H}_{19}\text{NO}_2$, the carbazole ring system is planar to within 0.032 (1) Å, and forms a dihedral angle of 74.42 (3)° with the plane of the attached benzene ring. Inversion-related molecules are linked by C—H... π hydrogen bonds into cyclic centrosymmetric dimers, which are interlinked into a chain along the *c* axis by π — π stacking interactions. Adjacent glide-related chains are interlinked via C—H... π interactions into a sheet parallel to the *bc* plane.

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Comment

Carbazole compounds are used as host materials for both small-molecule organic light-emitting diodes (OLEDs) and polymer OLEDs, due to their high triplet energy and good hole-transporting capability (Brunner *et al.*, 2004; van Dijken *et al.*, 2004). Carbazole derivatives exhibit antitumour (Leon *et al.*, 1988; Martin *et al.*, 2002; Routier *et al.*, 2005), antimycobacterial (Sunthitikawinsakul *et al.*, 2003), antifungal (Segall *et al.*, 2003) and potent anti-HIV (Yan *et al.*, 2005; Hirata *et al.*, 1999) activities. We report here the structure of the title compound, (I), a carbazole derivative.



The carbazole ring system in (I) (Fig. 1) is essentially planar, with a mean deviation of 0.018 Å. The dihedral angle between the C14—C19 benzene ring and the plane of the carbazole ring system is 74.42 (3)°. The C20—O1—C16—C17 [4.62 (16)°] and C21—O2—C19—C18 [−0.77 (16)°] torsion angles indicate that the two methoxy groups are coplanar with the C14—C19 benzene ring. Bond distances and angles in the carbazole rings are comparable with those reported for other carbazole derivatives (Duan *et al.*, 2006; Cui *et al.*, 2006; Wang *et al.*, 2006). The short H17...H20B (2.28 Å) contact causes the angles O1—C16—C17 [125.00 (11)°] and O1—C16—C15 [115.60 (10)°] to deviate significantly from 120°. In addition, the short H18...H21C (2.03 Å) contact results in the widening of the O2—C19—C18 angle to 124.97 (11)°.

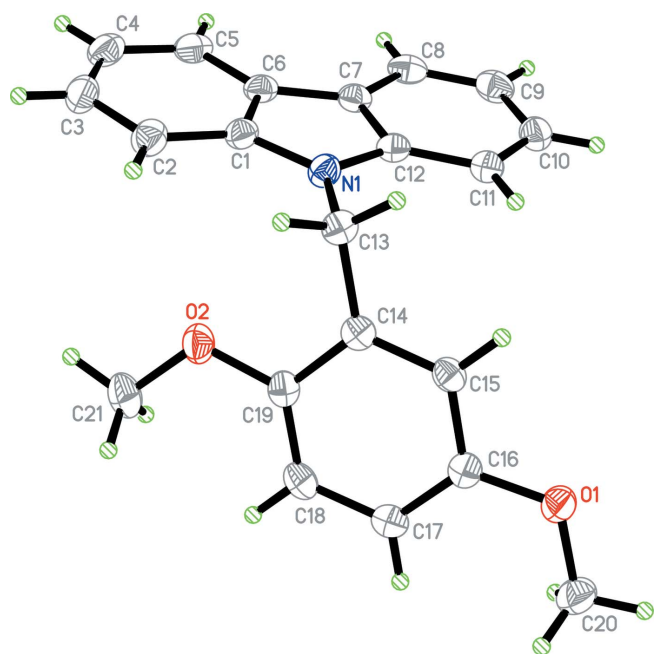


Figure 1
The molecular structure of (I), showing 60% probability displacement ellipsoids and the atomic numbering.

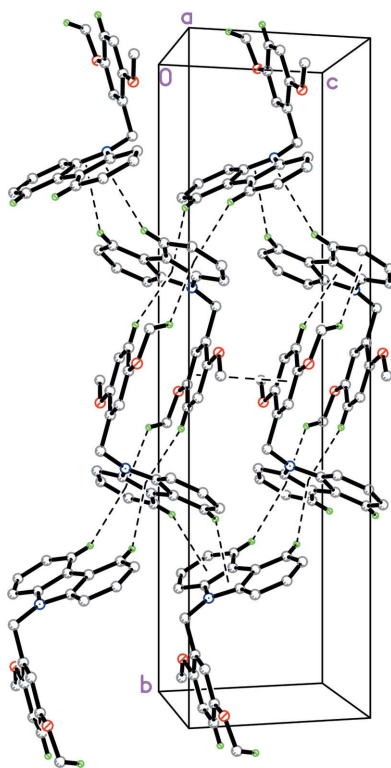


Figure 2
Part of a π - π and C-H... π hydrogen-bonded (dashed lines) sheet in (I). Only the H atoms involved in hydrogen bonding are shown.

In the crystal structure of (I), inversion-related molecules are linked by C17-H17...Cg1ⁱⁱ and C20-H20B...Cg2ⁱⁱ hydrogen bonds into cyclic centrosymmetric dimers (Cg1, Cg2 and symmetry codes are as defined in Table 1). The dimers are linked into a chain along the *c* axis by π - π stacking inter-

actions between the C14-C19 benzene rings of inversion-related molecules at (*x*, *y*, *z*) and ($-x$, $-y$, $-z$), whose centroids are separated by 3.6402 (6) Å. Adjacent glide-related chains are interlinked *via* C5-H5...Cg2ⁱ and C8-H8...Cg1ⁱ interactions (Table 1) into a sheet-like structure parallel to the *bc* plane (Fig. 2).

Experimental

Carbazole (1.50 mmol) was added to a suspension of NaH (12 mmol) in dry dimethylformamide (DMF; 10 ml) under nitrogen. The mixture was stirred for 15 min, after which time 2-bromomethyl-1,4-dimethoxybenzene (1.50 mmol) in dry DMF (10 ml) was added dropwise over a period of 30 min. The reaction mixture was refluxed overnight and then the addition of water (100 ml) caused precipitation of the crude product; the solid was filtered off and extracted with chloroform (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-chloroform (4:1 v/v). The compound was recrystallized from chloroform by slow evaporation.

Crystal data

C ₂₁ H ₁₉ NO ₂	<i>Z</i> = 4
<i>M_r</i> = 317.37	<i>D_x</i> = 1.306 Mg m ⁻³
Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Mo <i>K</i> α radiation
<i>a</i> = 9.2766 (1) Å	μ = 0.08 mm ⁻¹
<i>b</i> = 25.0761 (3) Å	<i>T</i> = 100 (2) K
<i>c</i> = 7.0947 (1) Å	Block, colourless
β = 101.935 (1)°	0.34 × 0.23 × 0.17 mm
<i>V</i> = 1614.70 (4) Å ³	

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	51344 measured reflections
ω scans	4935 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 2005)	3880 reflections with <i>I</i> > 2σ(<i>I</i>)
<i>T</i> _{min} = 0.938, <i>T</i> _{max} = 0.986	<i>R</i> _{int} = 0.045
	θ _{max} = 30.6°

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0743P)^2 + 0.2926P]$
$R[F^2 > 2\sigma(F^2)] = 0.046$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.137$	($\Delta\sigma$) _{max} = 0.001
<i>S</i> = 1.07	$\Delta\rho$ _{max} = 0.37 e Å ⁻³
4935 reflections	$\Delta\rho$ _{min} = -0.24 e Å ⁻³
219 parameters	
H-atom parameters constrained	

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 and Cg2 denote the centroids of the N1/C1/C6/C7/C12 and C1-C6 rings, respectively.

<i>D</i> -H... <i>A</i>	<i>D</i> -H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> -H... <i>A</i>
C5-H5...Cg2 ⁱ	0.95	2.78	3.6510 (14)	152
C8-H8...Cg1 ⁱ	0.95	2.74	3.5000 (14)	137
C17-H17...Cg1 ⁱⁱ	0.95	2.77	3.5808 (13)	144
C20-H20B...Cg2 ⁱⁱ	0.98	2.78	3.4752 (14)	129

Symmetry codes: (i) $x, -y - \frac{1}{2}, z + \frac{1}{2}$; (ii) $-x, -y, -z + 1$.

The H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C-H distances of 0.95 (aromatic), 0.98 (methyl) and 0.99 Å (methylene). The *U*_{iso}(H) values

were set equal to 1.2 or 1.5 (methyl) times U_{eq} of the carrier atoms. A rotating-group model was used for the methyl groups.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); 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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