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Ethyl 9-phenylsulfonylcarbazole-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.093
Data-to-parameter ratio = 20.7

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

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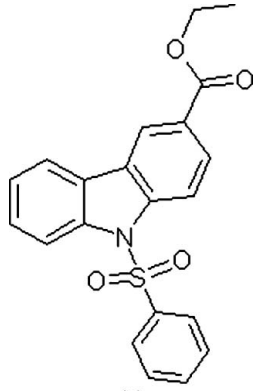
In the title compound, $C_{21}H_{17}NO_4S$, all bond lengths and angles show normal values. The sulfonyl-bound phenyl ring forms a dihedral angle of 88.31 (1)° with the mean plane of the carbazole fragment. The crystal packing is stabilized by weak intermolecular π–π, C—H···π and C—H···O interactions.

Comment

Carbazole compounds are used as host materials for organic light-emitting diodes (Brunner *et al.*, 2004; van Dijken *et al.*, 2004). They also exhibit antitumour (Leon *et al.*, 1988; Martin *et al.*, 2002; Routier *et al.*, 2005), antifungal (Segall *et al.*, 2003), antimicrobial (Martin & Prasad, 2006) and potent anti-HIV (Hirata *et al.*, 1999; Yan *et al.*, 2005) activities. We report here the structure of the title compound, (I) (Fig. 1).



(I)

The bond lengths and angles in (I) are normal and comparable to those reported for similar carbazole compounds (Govindasamy *et al.*, 1997*a*,*b*). The carbazole fragment is essentially planar with a mean deviation of 0.020 (1) Å. The sum of the bond angles around N1 [355.69°] indicates sp^2-hybridization. However, N1 deviates by 0.180 (1) Å from the S1/C1/C12 plane, suggesting some degree of pyramidalization.

Atom S1 has a distorted tetrahedral environment, with the angles O2—S1—O1 [120.11 (5)°] and N1—S1—C13 [105.11 (5)°] deviating significantly from the ideal tetrahedral values. These deviations are probably caused by the repulsive interactions between the short S═O bonds. The orientation of the phenylsulfonyl group with respect to the carbazole fragment is characterized by the torsion angles O1—S1—N1—C12 = 35.55 (10)°, O2—S1—N1—C1 = −41.32 (10)° and N1—S1—C13—C14 = 84.56 (9)°. The dihedral angles between the mean planes C13–C18 and N1/C1–C12, and O3/O4/C19/C20 and N1/C1–C12, are 88.31 (1) and 4.92 (6)°, respectively.

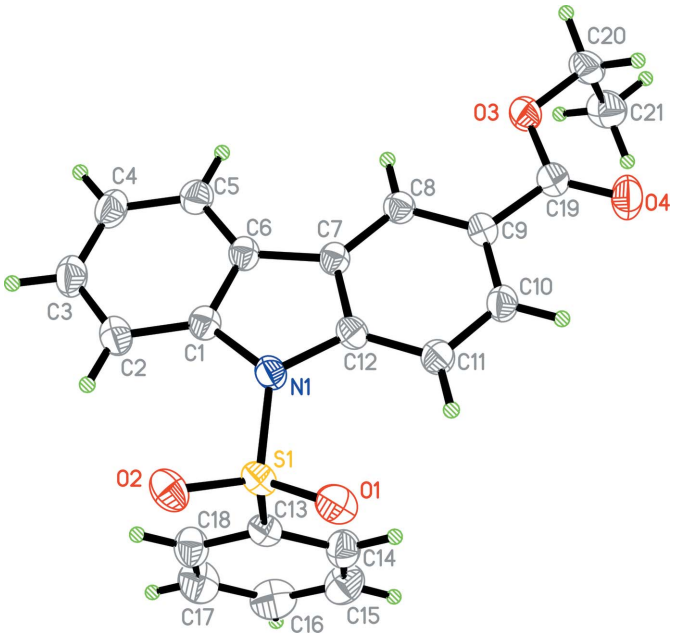


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 80% probability displacement ellipsoids.

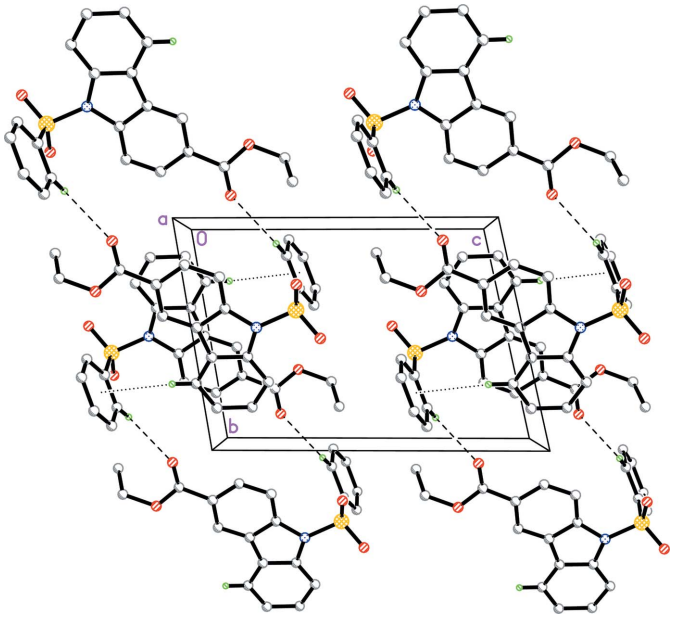


Figure 2
The crystal packing of (I) viewed along the *a* axis. The intermolecular C—H···O hydrogen bonds are shown as dashed lines. The dotted lines indicate C—H···π interactions. The H atoms not involved in hydrogen bonds and π–π interactions have been omitted for clarity.

In the crystal structure, the molecules are stacked into columns along the *a* axis, demonstrating C—H···π and π–π interactions. The former interaction involves the C13–C18 ring (centroid *Cg*1; Table 1), and the latter ones involve the rings N1/C1/C6/C7/C12 (centroid *Cg*2) and C7–C12 (centroid *Cg*3) with the short distances *Cg*2···*Cg*3^{iii} of 3.5891 (7) Å and *Cg*3···*Cg*3^{iii} of 3.5804 (6) Å [symmetry code: (iii) $2-x, 1-y, -z$]. The crystal packing (Fig. 2) is further stabilized by weak intermolecular C—H···O hydrogen bonds (Table 1), which generate $R_2^2(22)$ dimers.

Experimental

To a stirred solution of ethyl 3′-[1-phenylsulfonyl-2-methylindol-3-yl)acrylate (0.5 g, 1.35 mmol) in dry dimethylformamide (1.5 ml), dimethylacetamide (322 mg, 2.71 mmol) was added. The reaction mixture was heated at 383 K under N_2 for 3 h. It was then poured into 2% aqueous HCl (15 ml) and extracted with $CHCl_3$ (2 × 20 ml). The combined extracts were washed with water (10 ml) and brine (10 ml) and dried (Na_2SO_4). Removal of solvent followed by column chromatographic purification (silica gel, EtOAc–hexane 1:5) afforded compound (I) as a colourless solid (0.38 g, 73%). It was recrystallized from ethyl acetate and hexane (1:5 *v*/*v*).

Crystal data

$C_{21}H_{17}NO_4S$
M_r = 379.42
Triclinic, $P\overline{1}$
a = 8.0912 (2) Å
b = 9.1704 (2) Å
c = 12.3718 (2) Å
α = 78.685 (1)°
β = 87.567 (1)°
γ = 74.694 (1)°
V = 868.17 (3) Å^3
Z = 2
D_x = 1.451 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 100.0 (1) K
Plate, colourless
0.41 × 0.28 × 0.09 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.817, T_{max} = 0.980
27950 measured reflections
5056 independent reflections
4563 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 30.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.093
S = 1.02
5056 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.3804P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···*Cg*1^{i}	0.95	2.91	3.8605 (12)	174
C14—H14···O4^{ii}	0.95	2.56	3.2826 (15)	133

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

The H atoms were positioned geometrically with C—H distances of 0.95 Å (aromatic), 0.98 Å (methyl) and 0.99 Å (methylene), and were treated as riding on their parent C atoms, with U_{iso}(H) values of $1.2U_{eq}$(C) [$1.5U_{eq}$(C_{methyl})].

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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References

Bruker (2005). *APEX2* (Version 1.27), *SAINT* (Version 7.12A) and *SADABS* (Version 2004/1). Bruker AXS Inc., Madison, Wisconsin, USA.

Brunner, K., van Dijken, A., Borner, H., Bastiaansen, J. J., Kiggen, N. M. & Langeveld, B. M. (2004). *J. Am. Chem. Soc.* **126**, 6035–6042.

Dijken, A. van, Bastiaansen, J. J., Kiggen, N. M., Langeveld, B. M., Rothe, C., Monkman, A., Bach, I., Stossel, P. & Brunner, K. (2004). *J. Am. Chem. Soc.* **126**, 7718–7727.

Govindasamy, L., Velmurugan, D., Ravikumar, K. & Mohanakrishnan, A. K. (1997*a*). *Acta Cryst.* C**53**, 771–773.

Govindasamy, L., Velmurugan, D., Ravikumar, K. & Mohanakrishnan, A. K. (1997*b*). *Acta Cryst.* C**53**, 929–931.

Hirata, K., Ito, C., Furukawa, H., Itoigawa, M., Cosentino, L. M. & Lee, K. H. (1999). *Bioorg. Med. Chem. Lett.* **9**, 119–122.

Leon, P., Garbay-Jaureguiberry, C., Lambert, B., Le Pecq, J. B. & Roques, B. P. (1988). *J. Med. Chem.* **31**, 1021–1026.

Martin, A. E. & Prasad, K. J. (2006). *Acta Pharm.* **56**, 79–86.

Martin, G., Cocca, C., Rivera, E., Cricco, G., Caro, R., Segall, A., Pappa, H., Casaubon, R., Pizzorno, M. T. & Bergoc, R. M. (2002). *J. Exp. Ther. Oncol.* **2**, 77–84.

Routier, S., Peixoto, P., Merour, J. Y., Coudert, G., Dias, N., Bailly, C., Pierre, A., Leonce, S. & Caignard, D. H. (2005). *J. Med. Chem.* **48**, 1401–1413.

Segall, A. I., Vitale, M. F., Perez, V. L. & Pizzorno, M. T. (2003). *J. Pharm. Biomed. Anal.* **31**, 1021–1026.

Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Yan, H., Mizutani, T. C., Nomura, N., Takakura, T., Kitamura, Y., Miura, H., Nishizawa, M., Tatsumi, M., Yamamoto, N. & Sugiura, W. (2005). *Antivir. Chem. Chemother.* **16**, 363–373.