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2,3-Dibenzyl-1-(phenylsulfonyl)-1*H*-indole

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.099
Data-to-parameter ratio = 15.1

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

In the crystal structure of the title compound, $C_{28}H_{23}NO_2S$, the two benzyl groups are rotated in a such a way as to minimize their mutual interactions and their interaction with the phenylsulfonyl ring.

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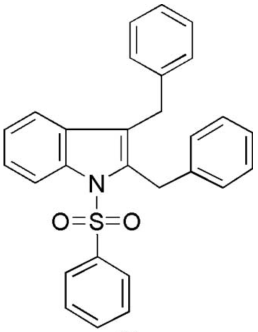
Comment

In connection with our interest in novel indole chemistry (Gribble *et al.*, 2005), we have synthesized the title compound, (I), and determined its crystal structure (Fig. 1). The observed parameters for (I) (Table 1) are comparable to the reported values for other 1-(phenylsulfonyl)indoles (Beddoes *et al.*, 1986; Schollmeyer *et al.*, 1995; Yokum & Fronczek, 1997; Govindasamy *et al.*, 1998; Sankaranarayanan *et al.*, 2000; Sonar *et al.*, 2004; Palani *et al.*, 2006*a*, *b*). The sum of the angles around the indole N atom is 352.1°, indicating significant pyramidalization of the nitrogen. The sulfonyl group is slightly twisted from the usual nitrogen–sulfonyl geometry seen in other 1-(phenylsulfonyl)indoles, in which the nitrogen lone pair eclipses the sulfonyl group (Beddoes *et al.*, 1986). Accordingly, in (I) the O1—S1—N1—C2 torsion angle is −38.3 (5)°. For comparison, the corresponding O1—S1—N1—C2 torsion angle in 1-(phenylsulfonyl)indole is −37° (Beddoes *et al.*, 1986) and in 2,3-dimethyl-1-(phenylsulfonyl)indole this angle is −36.4° (Palani *et al.*, 2006*b*).


(I)

Experimental

Potassium hydride (0.62 g of 30 wt% mineral oil dispersion, 15 mmol) was rinsed with hexanes (3 × 10 ml). The residual solvent was pumped off with a vacuum pump. Anhydrous THF (20 ml) was added *via* a cannula. The white suspension was cooled to 273 K. A red–brown solution of 2,3-dibenzylindole (0.92 g, 3.1 mmol) (Appleton *et al.*, 1993) in anhydrous THF (20 ml) was added slowly *via* a cannula. After 1 h benzenesulfonyl chloride (0.40 ml, 3.1 mmol) was added, using a syringe, to the red–brown reaction mixture. The reaction mixture turned orange. It was stirred for 36 h after which time it was poured on to crushed ice (50 ml). The organic layer was extracted with dichloromethane (3 × 25 ml), washed with distilled water (30 ml) and brine (30 ml), and dried with anhydrous magnesium sulfate. The solvent was evaporated *in vacuo*, yielding a brown oil, which was purified *via* column chromatography (4:1 hexanes–ethyl acetate). A greenish solid was obtained which was recrystallized from 1:1 dichloromethane/hexanes to give (I) as a yellow solid (0.60 g, 1.4 mmol, 45%; m.p. 395–397 K). HRMS calculated for $C_{28}H_{23}NO_2S$: 437.1450; found: 437.1444. Crystals suitable for X-ray determination were afforded by crystallization from diethyl ether–hexane (1:1).

Crystal data

$C_{28}H_{23}NO_2S$
$M_r = 437.56$
Monoclinic, $P2_1/n$
$a = 11.3830$ (4) Å
$b = 15.6589$ (6) Å
$c = 12.2180$ (5) Å
$\beta = 91.6083$ (18)°
$V = 2176.94$ (14) Å^3
$Z = 4$
$D_x = 1.335$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.18$ mm^{-1}
$T = 120$ K
Prism, colorless
0.38 × 0.36 × 0.34 mm

Data collection

Bruker–Nonius APEX2 diffractometer
φ and ω scans
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
$T_{\min} = 0.939$, $T_{\max} = 0.942$
27006 measured reflections
6427 independent reflections
4354 reflections with $I > 3\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\max} = 30.2°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.099$
$S = 1.05$
4354 reflections
289 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.72P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.72$ e Å^{-3}
$\Delta\rho_{\min} = -0.39$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.4282 (13)	C2—C3	1.353 (2)
S1—O2	1.4263 (13)	C2—C16	1.497 (2)
S1—N1	1.6626 (15)	C3—C4	1.445 (2)
S1—C10	1.7574 (16)	C3—C23	1.505 (2)
N1—C2	1.438 (2)	C16—C17	1.517 (2)
N1—C9	1.423 (2)	C23—C24	1.519 (2)
O1—S1—O2	119.79 (8)	C2—C3—C4	108.38 (15)
O1—S1—N1	106.23 (7)	C2—C3—C23	127.39 (16)
O2—S1—N1	107.17 (7)	C4—C3—C23	124.21 (16)
O1—S1—C10	108.57 (8)	C3—C4—C5	132.07 (17)
O2—S1—C10	108.68 (8)	C3—C4—C9	108.06 (15)
N1—S1—C10	105.50 (7)	N1—C9—C4	107.31 (14)
S1—N1—C2	124.50 (12)	N1—C9—C8	130.83 (16)
S1—N1—C9	120.28 (11)	S1—C10—C11	118.92 (13)
C2—N1—C9	107.36 (13)	S1—C10—C15	119.76 (13)
N1—C2—C3	108.80 (15)	C23—C24—C25	119.39 (15)
N1—C2—C16	123.55 (15)	C23—C24—C29	122.12 (16)
C3—C2—C16	127.50 (16)		

H atoms were included in the riding model approximation, with C—H = 1.00 Å, and with $U_{\rm iso}({\rm H}) = 1.17–1.22U_{\rm eq}({\rm C})$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT-NT* (Bruker, 2006); data reduction: *SAINT-NT*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CRYSTALS* (Betteridge *et al.*, 2003).

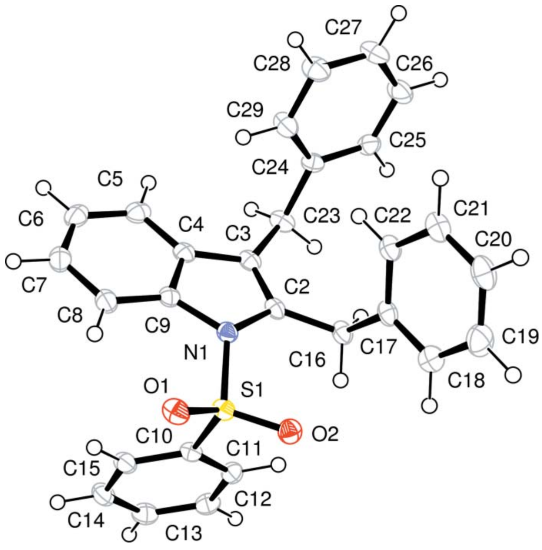


Figure 1
The molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms are drawn as spheres of arbitrary radius.

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