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1,2-Bis[1-phenylsulfonyl-3-(phenylthio)-indol-2-yl]ethene

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.120
Data-to-parameter ratio = 17.6

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

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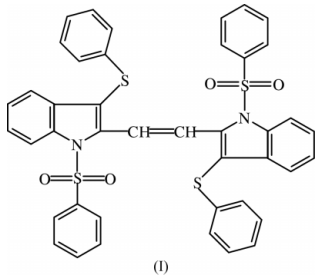
The title compound, $C_{42}H_{30}N_2O_4S_4$, crystallizes in the space group $P\overline{1}$ with half a molecule in the asymmetric unit and the other half generated by an inversion centre. The indole moiety is planar within ±0.043 (1) Å. The dihedral angle between the indole system and the thiophenyl ring is 83.4 (1)°. The S atom of the sulfonyl substituent has a distorted tetrahedral geometry. The molecular structure is stabilized by C—H···O and C—H···S interactions and the packing of the molecules in the solid state is stabilized by C—H···O, C—H···π and π–π intermolecular interactions.

Comment

The indole ring system is present in a number of natural products, many of which are found to possess antibacterial (Okabe & Adachi, 1998), antitumour (Schollmeyer *et al.*, 1995), antidepressant (Grinev *et al.*, 1984), antimicrobial (El-Sayed *et al.*, 1986; Gadaginamath & Patil, 1999) and anti-inflammatory (Rodriguez *et al.*, 1985) activities. Phenylsulfones show fungicidal activity comparable to or better than commercial fungicides (Wolf, 1999). The interaction of phenylsulfonyl indole with the calf-thymus DNA has also been studied by spectroscopic methods (Sivaraman *et al.*, 1996). Indoles have been proved to display high aldose reductase inhibitory activity (Rajeswaran *et al.*, 1999). The structure determination of the title compound, (I), was undertaken as part of our studies on indole derivatives.



(I)

The asymmetric unit of (I) contains one-half molecule with the other half generated by a centre of inversion; the centre of inversion lies at the midpoint of the C21—C21^{i} bond [symmetry code: (i) $1-x$, $1-y$, $-z$]. The bond distance C21—C21^{i} of 1.344 (3) Å confirms its double-bond character. The dihedral angle between the fused benzene and pyrrole rings is 3.9 (1)°, and atoms S1, S2 and C21 deviate by −0.702 (1), 0.009 (1) and 0.223 (2) Å, respectively, from the mean plane of the indole moiety. The sulfonyl phenyl ring (*A*) and thiophenyl ring (*B*) make dihedral angles of 70.1 (1) and 83.4 (1)°, respectively, with the indole moiety; rings *A* and *B* are inclined at an angle of 80.1 (1)°. The torsion angle O2—S1—N1—C1 of 169.6 (1)° and O2—S1—C9—C10 of

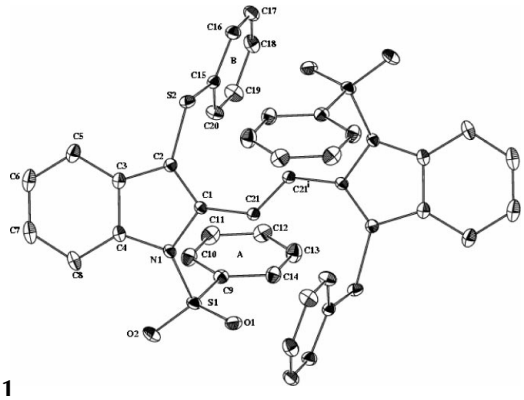


Figure 1
The molecular structure of (I), showing 35% probability displacement ellipsoids and the atom-numbering scheme.

−43.3 (2)° describe the conformation of the phenylsulfonyl group with respect to the indole system.

Atom S1 has a distorted tetrahedral geometry with the angles O1—S1—O2 [120.04 (8)°] and N1—S1—C9 [102.53 (7)°] deviating significantly from the regular tetrahedral value. The sum of the angles around atom N1 [351.4 (1)°] and the deviation of atom N1 by 0.257 (1) Å from the plane passing through atoms C1, C4 and S1 indicate slight pyramidalization of that atom. The bond angle C2—S2—C15 [102.69 (7)°] is reduced from 120°. The relatively large values of the C—N distances in the indole moiety [N1—C4 = 1.420 (2) Å and N1—C1 = 1.434 (2) Å] are due to the electron-withdrawing character of the phenylsulfonyl group. Similar features have also been observed for 1,2-bis(1-phenylsulfonyl-3-phenylthioindol-2-yl)ethane (SethuSankar *et al.*, 2002).

The orientation of the indole substituent is influenced by the weak C8—H8···O2 interaction, while the orientation of the sulfonylphenyl ring is conditioned by the weak C14—H14···O1 interaction. The torsion angles O2—S1—C9—C14, N1—S1—C9—C14, O1—S1—N1—C4 and S1—N1—C4—C8 (Table 1) define these orientations quantitatively. The molecular structure is stabilized by C—H···O and C—H···S interactions (Table 2). In the crystal, the indole moieties of the molecules at (x, y, z) and ($1-x$, $2-y$, $-z$) are stacked, such that the centroids of the pyrrole and benzene rings are separated by 3.685 (1) Å. Also, ring A of the inversion-related molecules (symmetry code: $-x$, $1-y$, $1-z$) are stacked with their centroids separated by 3.740 (1) Å. Apart from these weak π–π interactions, the packing of the molecules in the solid state is also stabilized by C—H···O and C—H···π intermolecular interactions.

Experimental

To a solution of *n*-butyllithium (15% solution in *n*-hexane) (0.9 ml, 2 mmol) in tetrahydrofuran (5 ml) at 273 K under nitrogen was added diethyl 1-benzenesulfonyl-3-phenylthioindol-2-ylmethyl phosphonate (1 g, 2 mmol) in the same solvent (40 ml), and the resulting solution stirred for 30 min. Dry oxygen was bubbled into the solution at 195 K for 30 min and at room temperature for 1 h with continued stirring. The reaction mixture was poured into ice water (40 ml). A sticky matter was extracted with methylene chloride (2 × 20 ml) and the extract was washed with water (2 × 10 ml) and dried ($MgSO_4$). The thick liquid obtained after removal of the solvent was crystallized from methanol, to afford the title compound as a crystalline solid. The melting point of the compound is 467 K.

Crystal data

$C_{42}H_{30}N_2O_4S_4$	$Z = 1$
$M_r = 754.92$	$D_x = 1.427$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 9.2920$ (2) Å	Cell parameters from 5090 reflections
$b = 9.5524$ (3) Å	$\theta = 2.3$–$28.3°$
$c = 11.3921$ (3) Å	$\mu = 0.32$ mm^{-1}
$\alpha = 67.379$ (1)°	$T = 293$ (2) K
$\beta = 72.821$ (1)°	Block, colourless
$\gamma = 74.589$ (1)°	0.46 × 0.40 × 0.32 mm
$V = 878.37$ (4) Å^3	

Data collection

Siemens SMART CCD area-detector diffractometer	3499 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.023$
ω scans	$\theta_{max} = 28.3°$
Absorption correction: none	$h = -10 \rightarrow 12$
6040 measured reflections	$k = -12 \rightarrow 12$
4138 independent reflections	$l = -15 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0767P)^2 + 0.0306P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.040$	
$wR(F^2) = 0.120$	
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.001$
4138 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
235 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.4244 (12)	S2—C15	1.7839 (15)
S1—O2	1.4268 (12)	N1—C4	1.4204 (18)
S1—N1	1.6943 (13)	N1—C1	1.4343 (16)
S1—C9	1.7635 (17)	C1—C21	1.4578 (18)
S2—C2	1.7502 (15)	C21—C21^{i}	1.344 (3)
O1—S1—O2	120.04 (8)	C2—S2—C15	102.69 (7)
O1—S1—N1	107.77 (7)	C4—N1—C1	108.05 (12)
O2—S1—N1	106.17 (7)	C4—N1—S1	122.14 (10)
O1—S1—C9	109.31 (7)	C1—N1—S1	121.22 (10)
O2—S1—C9	109.55 (8)	C2—C1—N1	107.87 (12)
N1—S1—C9	102.53 (7)		
O1—S1—N1—C4	155.46 (11)	N1—S1—C9—C14	−109.72 (14)
O2—S1—N1—C1	169.62 (11)	O2—S1—C9—C10	−43.28 (15)
C15—S2—C2—C1	−84.02 (14)	C2—S2—C15—C20	−3.92 (15)
S1—N1—C4—C8	−35.5 (2)	C2—C1—C21—C21^{i}	23.6 (3)
O2—S1—C9—C14	137.84 (14)	N1—C1—C21—C21^{i}	−163.16 (18)

Symmetry code: (i) $1-x, 1-y, -z$.

Table 2
Hydrogen-bonding geometry (Å, °).

$Cg(B)$ is the centroid of ring B.

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O2	0.93	2.33	2.869 (2)	117
C14—H14···O1	0.93	2.55	2.924 (2)	105
C21—H21···O1	0.93	2.37	2.984 (2)	123
C21—H21···S2^{i}	0.93	2.67	3.328 (2)	129
C17—H17···O2^{ii}	0.93	2.59	3.242 (2)	128
C6—H6···$Cg(B^{iii})$	0.93	2.99	3.842 (2)	153

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

All the H atoms were fixed geometrically and allowed to ride on their carrier atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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