Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

1,2-Bis(1-phenylsulfonyl-3-phenylthioindol-2-yl)ethane

K. SethuSankar,[a] N. Suresh Babu,[b] D. Velmurugan,[a]* S. Shanmuga Sundara Raj[c] and H.-K. Fun[c]

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [b]Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India, and [c]School of Physics, Universiti sains Malaysia, 11800 USM Penang, Malaysia

Correspondence e-mail: d velu@yahoo.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.056
wR factor = 0.144
Data-to-parameter ratio = 19.6

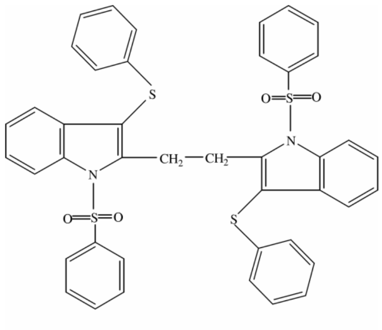
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 3 January 2002
Accepted 7 January 2002
Online 11 January 2002

In the title compound, $C_{42}H_{32}N_2O_4S_4$, the dihedral angle between the fused rings is 1.4 (1)°. The phenyl ring of the sulfonyl substituent makes a dihedral angle of 75.7 (1)° and the phenyl ring attached to the sulfide S atom makes a dihedral angle of 70.6 (1)° with the best plane of the indole ring. The molecular structure is stabilized by C—H···O hydrogen bonds.

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), antidepressive (Papenstasion & Newmeyer, 1972), antimicrobial (EI-Sayed *et al.*, 1986; Gadaginamath & Patil, 1999) and anti-inflammatory activities (Rodriguez *et al.*, 1985; Polletto *et al.*, 1974), and anti-implantation activity in rats. Indoles also intercalate with DNA (Sivaraman *et al.*, 1996). Intercalation between the base pairs in DNA has been implicated for their medicinal activities. The indole ring system occurs in plants (Nigović *et al.*, 2000). Indoles have been proved to display high aldose reductose inhibitory activity (Rajeswaran *et al.*, 1999). In order to obtain detailed information on the molecular conformation in the solid state, the X-ray study on an indole derivative has been carried out.



(I)

The asymmetric unit contains half a molecule and the centre of inversion lies in the C1—C1$^{\rm i}$ bond [symmetry code: (i) $-x$, $-y+1$, $-z$]. The bond distance C1—C1$^{\rm i}$ of 1.542 (5) Å confirms the single-bond character. Fig. 1 shows the *ORTEP*-3 plot (Farrugia, 1999) along with the atom-numbering scheme. Atom N1 deviates by 0.254 (2) Å from the plane passing through atoms S1, C9 and C2, indicating pyramidalization. Slight pyramidalization is also observed in related indoles (Yokum & Fronczek, 1997; Beddoes *et al.*, 1986). The torsion angles O2—S1—N1—C9 of −179.1 (2) and O2—S1—C10—C15 of −47.8 (3)° describe the conformation of the phenyl-

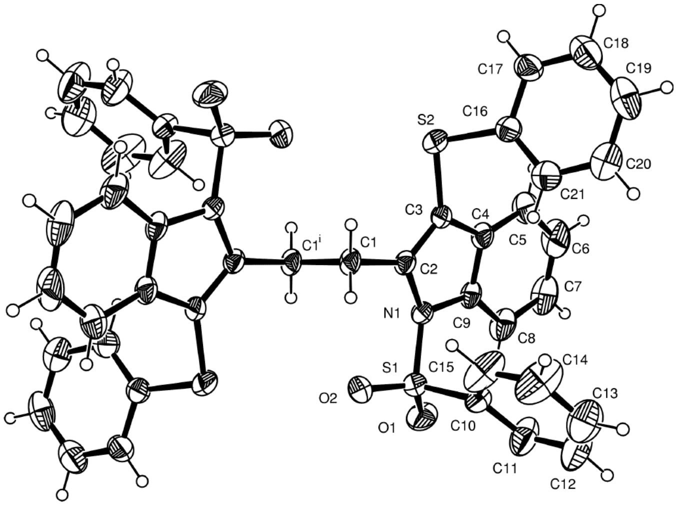


Figure 1
The molecular structure of the title compound showing 35% probability displacement ellipsoids and the atom-numbering scheme.

sulfonyl group with respect to the indole system, which causes the best planes of the indole and phenyl rings to form a dihedral angle of 75.7 (1)°, as observed in similar structures (Yokum & Fronzeck, 1997; Sankaranarayanan *et al.*, 2000). The dihedral angle between the indole ring system and the phenylthio group is 70.6 (1)°.

Atom S1 has a distorted tetrahedral geometry. The widening of the O1—S1—O2 angle to 120.0 (2)° and the resulting narrowing of the C10—S1—N1 angle to 104.2 (1)° from the ideal tetrahedral value is attributed to a Thorpe–Ingold effect (Bassindale, 1984). The S1—O1, S1—C10 and S1—N1 bond distances of 1.425 (2), 1.758 (3) and 1.671 (2) Å, respectively, are comparable with the reported values of 1.427 (4), 1.758 (1) and 1.642 (2) Å (Seetharaman & Rajan, 1995; Allen *et al.*, 1987). Atoms C1 and S2 deviate by 0.101 (1) and 0.083 (1) Å, respectively, from the mean plane passing through the indole moiety. The angle C3—S2—C16 of 101.8 (1)° is markably smaller than 120°. The relatively large values of the C—N distances in the indole moiety [N1—C9 1.423 (3) Å and N1—C2 1.442 (3) Å] are due to the electron-withdrawing character of the phenylsulfonyl group (Govindasamy *et al.*, 1997, 1998).

The C21—C16—S2 angle of 124.6 (2) is wider than C17—C16—S2 at 116.5 (2)°. The orientation of the indole substituent is influenced by a weak C8—H8···O1 interaction, while the orientation of the phenyl bound to the sulfonyl group is governed by the C11—H11···O1 interaction. The torsion angles O1—S1—C10—C11 of 2.4 (3), N1—S1—C10—C11 of −110.5 (3), O2—S1—N1—C9 of −179.1 (2) and S1—N1—C9—C8 of 28.8 (4)° quantitatively define these orientations. The molecular structure is stabilized by C—H···O hydrogen bonds (Table 2).

Experimental

The title compound was obtained by reductive debromination of *N*-phenylsulfonyl-2-bromomethyl-3-phenylthioindole by prolonged heating with indium foil in dry tetrahydrofuran.

Crystal data

$C_{42}H_{32}N_2O_4S_4$
$M_r = 756.94$
Monoclinic, $P2_1/n$
$a = 12.5439$ (4) Å
$b = 8.3686$ (2) Å
$c = 18.9898$ (6) Å
$\beta = 106.881$ (1)°
$V = 1907.55$ (10) Å^3
$Z = 2$
$D_x = 1.318$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 13390 reflections
$\theta = 1.7$–28.3°
$\mu = 0.29$ mm^{-1}
$T = 293$ (2) K
Needle, colourless
0.46 × 0.16 × 0.12 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
12921 measured reflections
4615 independent reflections
2139 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.086$
$\theta_{\rm max} = 28.3°$
$h = -13 \rightarrow 16$
$k = -10 \rightarrow 11$
$l = -24 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.144$
$S = 0.93$
4615 reflections
236 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.31$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0046 (8)

Table 1
Selected geometric parameters (Å, °).

S2—C3	1.754 (3)	N1—C9	1.423 (3)
S2—C16	1.774 (3)	N1—C2	1.442 (3)
O2—S1—N1	106.5 (1)	C9—N1—C2	107.5 (2)
O1—S1—N1	106.1 (1)	C2—N1—S1	123.7 (2)
O2—S1—C10	110.0 (2)	C3—C2—N1	107.5 (2)
O1—S1—C10	108.8 (1)	C3—C2—C1	127.9 (2)
N1—S1—C10	104.2 (1)		
C10—S1—N1—C9	64.7 (2)	N1—C2—C3—S2	175.1 (2)
C10—S1—N1—C2	−79.5 (2)	S1—N1—C9—C8	28.8 (4)
C9—N1—C2—C3	2.2 (3)	S1—N1—C9—C4	−151.8 (2)
S1—N1—C2—C3	150.3 (2)	O2—S1—C10—C11	135.7 (3)
S1—N1—C2—C1	−36.8 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···S2	0.97	2.85	3.292 (3)	109
C1—H1*B*···O2	0.97	2.30	2.857 (3)	115
C8—H8···O1	0.93	2.41	2.985 (4)	120
C11—H11···O1	0.93	2.54	2.913 (4)	105

All the H atoms were geometrically fixed and allowed to ride on the corresponding non-H 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: *ORTEP*-3 for Windows (Farrugia, 1999); software used to prepare

Acta Cryst. (2002). E**58**, o134–o136 K. Sethu Sankar *et al.* • $C_{42}H_{32}N_2O_4S_4$ **o135**

material for publication: *SHELXL*97 and *PARST* (Nardelli, 1983, 1995).

DV thanks DST, India. SSSR thanks Universiti Sains Malaysia for a visiting Postdoctoral Research Fellowship and HKF would like to thank the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 190-9609-2801. The authors thank Professor P. C. Srinivasan for providing the sample for X-ray study.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* pp. S1–19.

Bassindale, A. (1984). *The Third Dimension in Organic Chemistry*, ch 1, p. 11. New York: John Wiley and Sons.

Beddoes, R. L., Dalton, L., Joule, J. A., Mills, O. S., Street, J. O. & Watt, C. I. F. (1986). *J. Chem. Soc. Perkin Trans.* 2, pp. 787–797.

El-Sayed, K., Barnhart, D. M., Ammon, H. L. & Wassel, G. M. (1986). *Acta Cryst.* C**42**, 1383–1385.

Farrugia, L. J. (1999). *ORTEP*-3 for Windows. Version 1.05. University of Glasgow, Scotland.

Gadaginamath, G. S. & Patil, S. A. (1999). *Indian J. Chem. Sect. B*, **38**, 1070–1074.

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

Govindasamy, L. Velmurugan, D., Ravikumar, K. & Mohanakrishnan, A. K. (1998). *Acta Cryst.* C**54**, 635–637.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Nigović, B., Antolic, S., Kojic Prodic, B., Kiralj, R., Magnus, V. & Salopek-Sondi, B. (2000). *Acta Cryst.* B**56**, 94–111.

Okabe, N. & Adachi, Y. (1998). *Acta Cryst.* C**54**, 386–387.

Papenstasion, Z. W. & Newmeyer, J. L. (1972). US Patent 3, **674**, 809/1972; *Chem. Abstr.* **77**, 126425.

Polletto, J. P., Allen, G. R. & Weiss, M. J. (1974). US Patent 3801, 594; *Chem. Abstr.* **77**, 126425.

Rajeswaran, W. G., Labroo, R. B. & Cohen, L. A. (1999). *J. Org. Chem.* **64**, 1369–1371.

Rodriguez, J. G., Temprano, F., Esteban-Calderon, C., Martinez-Ripoll, M. & Garcia-Blanco, S. (1985). *Tetrahedron*, **41**, 3813–3823.

Sankaranarayanan, R., Velmurugan, D., Shanmuga Sundara Raj, S., Fun, H.-K., Babu, G. & Perumal, P. T. (2000). *Acta Cryst.* C**56**, 475–476.

Schollmeyer, D., Fischer, G. & Pindur, U. (1995). *Acta Cryst.* C**51**, 2572–2575.

Seetharaman, J. & Rajan, S. S. (1995). *Acta Cryst.* C**51**, 78–80.

Sivaraman, J., Subramanian, K., Velmurugan, D., Subramanian, E. & Seetharaman, J. (1996). *J. Mol. Struct.* **385**, 123–128.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Siemens (1996). *SMART* and *SAINT*. Version 4.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Yokum, S. T. & Fronczek, F. R. (1997). *Acta Cryst.* C**53**, 362–363.

o136 K. Sethu Sankar *et al.* • $C_{42}H_{32}N_2O_4S_4$ *Acta Cryst.* (2002). E**58**, o134–o136