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cis-3-(*p*-Bromostyryl)-2-methyl-*N*-(phenylsulfonyl)indole

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In the title compound, $C_{23}H_{18}BrNO_2S$, the bromophenyl group is oriented *cis* with respect to the indole moiety. The phenylsulfonyl group is nearly perpendicular to the indole ring system, forming a dihedral angle of 84.6 (1)°.

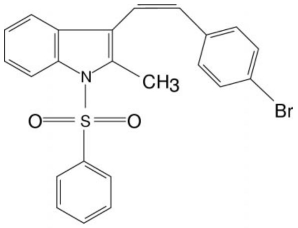
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.112
Data-to-parameter ratio = 17.3

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

Comment

Indole derivatives are pharmacologically active. 1,2,3,4-Tetrahydropiperazino[1,2-*a*]indoles and -diazepino[1,2-*a*]-indoles are found to possess central nervous system depressant (Yamamoto *et al.*, 1974), antihypertensive (Merck, 1973) and muscle-relaxant (Hendi & Basangoudar, 1981) properties. Spiro-indole derivatives exhibit antibacterial and antifungal properties (Sehgal *et al.*, 1994). These types of compounds are also used as anti-inflammatory (Rodriguez *et al.*, 1985) and antimicrobial (El-Sayed *et al.*, 1986) agents. 3-(Indol-3-yl)-2-oxopropanoic acid (indole pyruvic acid) is a well known endogenous metabolite of tryptophan aminotransferase. It has anxiolytic activity in mice, similar to that of diazepam (Lapin & Politi, 1993), and is known to regulate the modulation of brain kynurenic acid content. Some indole compounds have DNA intercalation properties (Sivaraman *et al.*, 1996). Indole-3-acetic acid is a plant growth regulator (Gilchrist, 1997) and 4-oxo-4,5,6,7-tetrahydro-3-indoleacetic acids display anti-diabetic properties (Nagarajan *et al.*, 1988). In this paper, we report the structure of the title compound, (I), an indole derivative.



(I)

Fig. 1 shows the molecular structure of (I), with the atom-numbering scheme. The observed bond distances compare well with previously reported values (Beddoes *et al.*, 1994; Seetharaman & Rajan, 1995; Singh *et al.*, 1985; Sivaraman *et al.*, 1994*b*). The sum of the angles around N1 [357.4 (2)°] indicates sp^2 hybridization. The sulfonyl group has a distorted tetrahedral geometry, with widening of the O1—S1—O2 [120.2 (1)°] angle and the resultant narrowing of the N1—S1—C9 [104.0 (1)°] angle from ideal tetrahedral values. Similar distortions in the sulfonyl group have been reported (Beddoes *et al.*, 1994; Govindasamy *et al.*, 1998; Govind *et al.*, 2002) and they are attributed to the Thorpe–Ingold effect (Bassindale,

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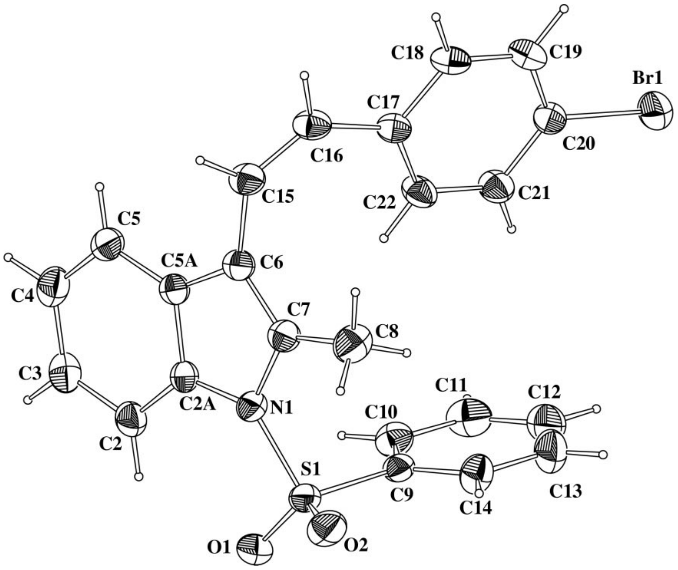

Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

1984). In the indole ring system, the C—C—C angles at C2 and C5 are narrowed to 117.5 (2) and 118.9 (2)°, respectively, while that at C2*A* is widened to 121.6 (2)°. Similar effects have been observed previously (Allen & Trotter, 1970; Sivaraman *et al.*, 1994*a*,*b*,*c*; Govind *et al.*, 2002) and they are attributed to the fusion of a smaller pyrrole ring to the six-membered benzene ring. As a result of steric interactions, the C16—C17—C22 angle [126.0 (2)°] is wider than the C16—C17—C18 angle [117.8 (2)°]. The indole moiety is nearly planar, with the fused benzene and pyrrole rings forming a dihedral angle of 3.8 (1)°. The phenyl ring attached to the sulfonyl group and the bromophenyl ring form dihedral angles of 84.6 (1) and 66.7 (1)°, respectively, with the indole moiety. The C6—C15—C16—C17 torsion angle of 4.2 (5)° indicates the *cis* orientation of the bromophenyl group with respect to the indole moiety. Short intramolecular C—H···O contacts involving the sulfonyl O atoms are observed (Table 2). A short intermolecular Br···O contact is observed between Br1 and O2(x, $1 + y$, $z - 1$) [3.362 (2) Å].

Experimental

To a suspension of NaH (0.15 g, 6 mmol) in dry tetrahydrofuran (20 ml) at room temperature was slowly added 4-bromophenylphosphonium bromide (6 mmol) in dry tetrahydrofuran (40 ml) under N_2, and the resulting solution was stirred for 1 h. A solution of 1-phenylsulfonyl-2-methylindole-3-carbaldehyde (1 equivalent, 6 mmol) in dry tetrahydrofuran (20 ml) was then added. Stirring was continued for another 2 h. The red–brown solution was poured over ice (200 g) containing saturated NH_4Cl solution. Then it was extracted with dichloromethane and the organic extract was washed with water (3 × 25 ml), dried over $MgSO_4$ and filtered. Removal of solvent and chromatographic separation on a silica-gel column by elution with hexane/ethyl acetate (9:1) afforded the the title vinylindole (m.p. 413 K). Single crystals were obtained by slow evaporation of a hexane–ethyl acetate (9:1) solution.

Crystal data

$C_{23}H_{18}BrNO_2S$
$M_r = 452.35$
Triclinic, $P\overline{1}$
$a = 10.2443$ (3) Å
$b = 10.4161$ (3) Å
$c = 11.4190$ (3) Å
$\alpha = 65.064$ (1)°
$\beta = 89.772$ (1)°
$\gamma = 65.707$ (1)°
$V = 985.65$ (5) Å^3

$Z = 2$
$D_x = 1.524$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3502 reflections
$\theta = 2.0$–27.5°
$\mu = 2.21$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.48 × 0.46 × 0.42 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.417$, $T_{\max} = 0.457$
6560 measured reflections

4420 independent reflections
3451 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 27.5°$
$h = -13 \rightarrow 12$
$k = -13 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.112$
$S = 1.03$
4420 reflections
255 parameters

H-atom parameters constrained
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.66$ e Å^{-3}
$\Delta\rho_{\min} = -0.68$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.015 (3)

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.424 (2)	N1—C7	1.421 (3)
S1—N1	1.673 (2)	C6—C15	1.472 (3)
S1—C9	1.756 (2)	C15—C16	1.330 (4)
N1—C2*A*	1.415 (3)	C16—C17	1.467 (3)
O2—S1—O1	120.2 (1)	C4—C5—C5*A*	118.9 (2)
N1—S1—C9	104.0 (1)	C6—C7—C8	127.7 (2)
C2*A*—N1—C7	108.4 (2)	N1—C7—C8	123.6 (2)
C2*A*—N1—S1	125.6 (2)	C16—C15—C6	129.4 (2)
C7—N1—S1	123.4 (2)	C15—C16—C17	131.9 (2)
C2*A*—C2—C3	117.5 (2)	C18—C17—C16	117.8 (2)
C2—C2*A*—C5*A*	121.6 (2)	C22—C17—C16	126.0 (2)
C6—C15—C16—C17	4.2 (5)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1	0.93	2.30	2.886 (5)	121
C8—H8*B*···O2	0.96	2.50	3.000 (5)	112
C14—H14···O2	0.93	2.52	2.898 (3)	105

All H atoms were fixed geometrically and allowed to ride on their parent C 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); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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