Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,5-Dimethyl-7-phenylsulfonyl-5,6-dihydroindolo[2,3-*c*]benzazepin-12-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.051
wR factor = 0.123
Data-to-parameter ratio = 16.3

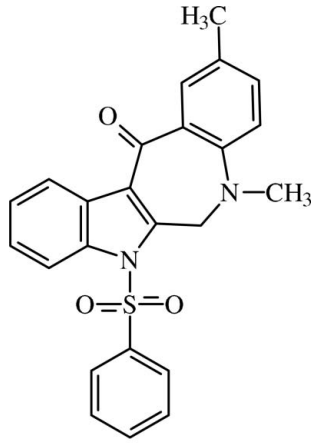
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{24}H_{20}N_2O_3S$, crystallizes with two independent molecules in the asymmetric unit, related by a non-crystallographic twofold rotation axis. The two molecules differ in the relative orientations of the phenylsulfonyl group and the indole ring system. In both molecules, the seven-membered ring adopts a distorted boat conformation. The molecular packing is stabilized by C—H···π and C—H···O interactions.

Received 15 June 2005
Accepted 30 June 2005
Online 6 July 2005

Comment

Indole derivatives have been found to exhibit antibacterial, antifungal (Wang & Ng, 2002; Singh *et al.*, 2000; Tsotinis *et al.*, 1997; Quetin-Leclercq *et al.*, 1995) and antitumour activities (Andreani *et al.*, 2001; Bradlow *et al.*, 1999; Cirrincione *et al.*, 1999; Tiwari *et al.*, 1994; Dashwood *et al.*, 1994). Certain indole derivatives are used as neuroprotectants (Stolc, 1999). Some of the indole alkaloids extracted from plants possess interesting cytotoxic, antitumour or antiparasitic properties (Quetin-Leclercq, 1994; Mukhopadhyay *et al.*, 1981). Pyrido[1,2-*a*]indole derivatives have been identified as potent inhibitors of human immunodeficiency virus type 1 (Taylor *et al.*, 1999), and 5-chloro-3-(phenylsulfonyl)indole-2-carboxamide is reported to be a highly potent non-nucleoside inhibitor of HIV-1 reverse transcriptase (Williams *et al.*,1993). The interaction of phenylsulfonylindole with calf thymus DNA has also been studied by spectroscopic methods (Sivaraman *et al.*, 1996). The structure determination of the title compound, (I), was undertaken as part of our investigations of indole derivatives.



(I)

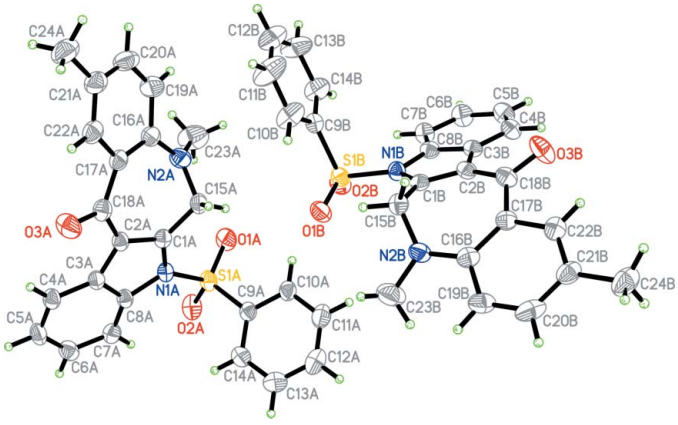


Figure 1
The asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

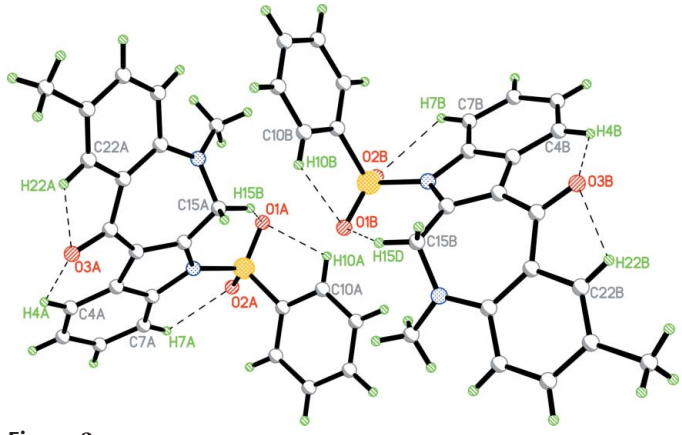


Figure 2
A view of the intramolecular interactions (dashed lines) in (I). Only the atoms involved in the interactions are labelled.

The asymmetric unit of (I) contains two crystallographically independent molecules, which are related by a local twofold rotation axis situated approximately parallel to [100]. A displacement ellipsoid plot of the two independent molecules, *A* and *B*, is shown in Fig. 1. The corresponding bond lengths of the two molecules agree with each other and a difference of 2° is observed in the S1—N1—C8 [125.8 (2) and 123.8 (2)°] and C2—C18—C17 [123.6 (3) and 121.6 (3)°] bond angles. Molecules *A* and *B* differ in the relative orientations of the phenylsulfonyl group and the indole ring system, as indicated by the torsion angles quoted in Table 1.

In both molecules, the indole ring system is planar (r.m.s deviation 0.021 Å for *A* and 0.009 Å for *B*) and atom N2 is in a planar configuration. The length of the N—Csp^2 bond in the seven-membered rings is close to the mean value of 1.355 (14) Å reported for N atoms with planar configurations (Allen *et al.*, 1987), whereas those in the pyrrole rings are longer (Table 1).

As a result of the repulsive interaction between the short S=O bonds, atom S1 has a distorted tetrahedral configuration, with O—S—O and N—S—C angles deviating significantly from ideal tetrahedral values. As observed in other related phenylsulfonylindoles (Ravishankar *et al.*, 2003*a*,*b*, 2005*a*,*b*), the orientation of the phenylsulfonyl group with respect to the indole ring system is influenced by intramolecular C—H···O interactions (Table 2) involving the sulfonyl atoms O1 and O2 (Fig. 2). These interactions generate rings of graph-set motif *S*(5) or *S*(6) (Bernstein *et al.*, 1995; Etter, 1990). The dihedral angle between the sulfonyl-bound phenyl ring and the indole ring system is 75.27 (9)° in *A* and 76.96 (12)° in *B*.

Least-squares plane calculations indicate that the seven-membered ring adopts a distorted boat conformation in both molecules. Atoms N2, C1, C2 and C16 are coplanar (r.m.s deviation 0.075 Å in *A* and 0.042 Å in *B*), and the deviations of atoms C15, C17 and C18 from the plane are 0.693 (4), 0.405 (5) and 0.341 (6) Å, respectively, in *A*, and 0.698 (4), 0.390 (5) and 0.413 Å, respectively, in *B*. The torsion angles within the seven-membered ring (Table 1) deviate significantly from the torsion angles of a cycloheptane ring adopting a boat conformation (Allen *et al.*, 1993). The C16/C17/C19–C22 benzene ring forms a dihedral angle of 35.69 (11)° [37.00 (11)° in *B*] with the mean plane through the indole ring system.

The two independent molecules are linked to form a three-dimensional network via C—H···π interactions (Table 2) involving atoms H11*B*, H12*B* and H24*D* of molecule *B*, the C3–C8 benzene ring of molecule *A* (centroid *Cg*2) and the C9–C14 phenyl ring of molecule *A* (centroid *Cg*1). In addition, the molecular packing is stabilized by intermolecular C—H···O interactions (Table 2).

Experimental

A mixture of 1-phenylsulfonyl-2-[2′-acetamido-5′-methylbenzoyl]-indole (700 mg, 0.16 mmol), chloromethyl methyl ether (20 ml) and acetic acid (20 ml) was stirred for 72 h at room temperature, then poured into ice–water and extracted with chloroform (100 ml). The residue was chromatographed over silica gel using 20% ethyl acetate in hexane as eluent, and crystallized by the slow evaporation method.

Crystal data

$C_{24}H_{20}N_2O_3S$
M_r = 416.48
Orthorhombic, $Pna2_1$
a = 15.6032 (7) Å
b = 15.9227 (7) Å
c = 16.3694 (7) Å
V = 4066.9 (3) Å^3
Z = 8
D_x = 1.360 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8072 reflections
θ = 2.2–28.3°
μ = 0.19 mm^{-1}
T = 293 (2) K
Block, yellow
0.50 × 0.40 × 0.40 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.912, T_{max} = 0.929
24897 measured reflections
8896 independent reflections
6111 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 28.3°
$h = -19 \rightarrow 20$
$k = -21 \rightarrow 20$
$l = -21 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2 + 0.5548P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.051$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.123$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
$S = 1.09$	$\Delta\rho_{min} = -0.29$ e Å^{-3}
8896 reflections	Absolute structure: Flack (1983), with 3692 Friedel pairs
545 parameters	Flack parameter: 0.01 (7)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1*A*—C1*A*	1.402 (4)	N1*B*—C1*B*	1.412 (4)
N1*A*—C8*A*	1.431 (4)	N1*B*—C8*B*	1.428 (4)
N2*A*—C16*A*	1.387 (4)	N2*B*—C16*B*	1.374 (4)
O1*A*—S1*A*—O2*A*	119.89 (15)	O1*B*—S1*B*—O2*B*	120.09 (14)
N1*A*—S1*A*—C9*A*	105.48 (13)	N1*B*—S1*B*—C9*B*	105.26 (13)
C1*A*—N1*A*—C8*A*	107.9 (2)	C1*B*—N1*B*—C8*B*	108.0 (2)
C1*A*—N1*A*—S1*A*	126.2 (2)	C1*B*—N1*B*—S1*B*	127.9 (2)
C8*A*—N1*A*—S1*A*	125.8 (2)	C8*B*—N1*B*—S1*B*	123.8 (2)
C2*A*—C18*A*—C17*A*	123.5 (3)	C2*B*—C18*B*—C17*B*	121.5 (3)
O1*A*—S1*A*—N1*A*—C1*A*	−35.9 (3)	O1*B*—S1*B*—N1*B*—C1*B*	−22.7 (3)
O2*A*—S1*A*—N1*A*—C1*A*	−164.8 (2)	O2*B*—S1*B*—N1*B*—C1*B*	−151.6 (2)
C9*A*—S1*A*—N1*A*—C1*A*	79.8 (3)	C9*B*—S1*B*—N1*B*—C1*B*	93.3 (3)
O1*A*—S1*A*—N1*A*—C8*A*	147.6 (2)	O1*B*—S1*B*—N1*B*—C8*B*	162.9 (2)
O2*A*—S1*A*—N1*A*—C8*A*	18.7 (3)	O2*B*—S1*B*—N1*B*—C8*B*	33.9 (3)
C9*A*—S1*A*—N1*A*—C8*A*	−96.7 (3)	C9*B*—S1*B*—N1*B*—C8*B*	−81.1 (3)
C15*A*—C1*A*—C2*A*—C18*A*	−0.1 (5)	C15*B*—C1*B*—C2*B*—C18*B*	3.3 (5)
C16*A*—N2*A*—C15*A*—C1*A*	79.2 (4)	C16*B*—N2*B*—C15*B*—C1*B*	75.5 (3)
C2*A*—C1*A*—C15*A*—N2*A*	−60.6 (4)	C2*B*—C1*B*—C15*B*—N2*B*	−65.3 (4)
C15*A*—N2*A*—C16*A*—C17*A*	−30.7 (5)	C15*B*—N2*B*—C16*B*—C17*B*	−23.8 (4)
N2*A*—C16*A*—C17*A*—C18*A*	−16.3 (5)	N2*B*—C16*B*—C17*B*—C18*B*	−16.6 (5)
C1*A*—C2*A*—C18*A*—C17*A*	28.0 (5)	C1*B*—C2*B*—C18*B*—C17*B*	31.1 (5)
C16*A*—C17*A*—C18*A*—C2*A*	2.2 (5)	C16*B*—C17*B*—C18*B*—C2*B*	−3.8 (5)

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

*Cg*1 is the centroid of the C9–C14 ring and *Cg*2 is the centroid of the C3–C8 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4*A*—H4*A*···O3*A*	0.93	2.55	3.047 (4)	114
C4*B*—H4*B*···O3*B*	0.93	2.57	3.054 (4)	113
C7*A*—H7*A*···O2*A*	0.93	2.29	2.873 (4)	121
C7*B*—H7*B*···O2*B*	0.93	2.39	2.940 (4)	118
C10*A*—H10*A*···O1*A*	0.93	2.51	2.884 (4)	104
C10*B*—H10*B*···O1*B*	0.93	2.55	2.912 (4)	103
C15*A*—H15*B*···O1*A*	0.97	2.30	2.997 (4)	128
C15*B*—H15*D*···O1*B*	0.97	2.24	2.959 (4)	130
C22*A*—H22*A*···O3*A*	0.93	2.33	2.694 (4)	103
C22*B*—H22*B*···O3*B*	0.93	2.32	2.682 (4)	103
C11*B*—H11*B*···*Cg*1^{i}	0.93	2.85	3.687 (5)	151
C12*B*—H12*B*···*Cg*2^{ii}	0.93	2.86	3.702 (5)	151
C24*B*—H24*D*···*Cg*1^{iii}	0.96	2.90	3.698 (5)	142

Symmetry codes: (i) $x+\frac{1}{2}, \frac{1}{2}-y, z$; (ii) $\frac{1}{2}-x, y-\frac{1}{2}, z+\frac{1}{2}$; (iii) $1-x, 1-y, z+\frac{1}{2}$.

H atoms were positioned geometrically and treated as riding on their parent C atoms, with C—H distances of 0.93 (aromatic), 0.97 (methylene) or 0.96 Å (methyl), and with $U_{iso}(H) = 1.2U_{eq}(C)$, or $1.5U_{eq}(C)$ for methyl H atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

HKF thanks the Malaysian Government and Universiti Sains Malaysia for research grant R&D No. 305/PFIZIK/610961.

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