$a = 7.666$ (3) Å
$b = 15.521$ (2) Å
$c = 7.470$ (2) Å
$\alpha = 101.53$ (2)°
$\beta = 115.50$ (3)°
$\gamma = 91.91$ (3)°
$V = 778.8$ (4) Å^3
$Z = 2$
$D_x = 1.460$ Mg m^{-3}
D_m not measured

$\theta = 10.86$–15.67°
$\mu = 0.334$ mm^{-1}
$T = 293$ (2) K
Plate
0.7 × 0.6 × 0.3 mm
Colourless

Data collection

Rigaku AFC-5*S* diffractometer
$2\theta/\omega$ scan
Absorption correction: analytical (de Meulenaer & Tompa, 1965)
$T_{\min} = 0.823$, $T_{\max} = 0.914$
11 308 measured reflections
5654 independent reflections
4593 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.012$
$\theta_{\max} = 32.57°$
$h = -11 \rightarrow 11$
$k = -23 \rightarrow 23$
$l = -11 \rightarrow 11$
3 standard reflections every 150 reflections intensity decay: <2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.119$
$S = 1.085$
5654 reflections
261 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.0863P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 0.514$ e Å^{-3}
$\Delta\rho_{\min} = -0.531$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.093 (7)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 3. *Selected geometric parameters (Å, °) for (II)*

P1—O13	1.575 (1)	C63—C5	1.491 (2)
P1—O11	1.570 (1)	N3—N4	1.386 (1)
P1—N3	1.647 (1)	N3—C31	1.446 (2)
P1—S2	1.911 (1)	O51—C5	1.221 (2)
O11—C12	1.438 (2)	C5—N4	1.350 (2)
O13—P1—O11	99.1 (1)	N4—N3—C31	115.7 (1)
O13—P1—N3	99.4 (1)	N4—N3—P1	120.4 (1)
O11—P1—N3	108.2 (1)	C31—N3—P1	122.2 (1)
O13—P1—S2	116.7 (1)	O51—C5—N4	122.9 (1)
O11—P1—S2	116.5 (1)	O51—C5—C63	122.5 (1)
N3—P1—S2	114.6 (1)	N4—C5—C63	114.6 (1)
C12—O11—P1	121.3 (1)	C5—N4—N3	120.7 (1)
C14—O13—P1	121.1 (1)		

Table 4. *Hydrogen-bonding geometry (Å, °) for (II)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H41···O641	0.84 (2)	2.02 (2)	2.684 (1)	135 (2)

For (I), atoms H141, H142 and H143 had U(H) fixed to $1.3U_{\rm eq}$ of their parent atoms. For both (I) and (II), the other methyl H atoms were constrained to their parent C atom by *DFIX* in *SHELXL*97 (Sheldrick, 1997) using a rigid body model. The remaining H atoms had all their parameters refined.

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989*a*); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989*b*); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*97; molecular graphics: *ORTEX* (McArdle, 1995). Software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1996) for (I); *SHELXL*97 for (II).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1410). Services for accessing these data are described at the back of the journal.

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Dimethyl α-(2-benzyl-1-methyl-4-phenylsulfonylpyrrolo[3,4-*b*]indol-3-yl)maleate

L. Govindasamy,[a] D. Velmurugan,[a] S. Shanmuga Sundara Raj[b] and Hoong-Kun Fun[b]

[a]*Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and* [b]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia. E-mail: crystal@giasmd01.vsnl.net.in*

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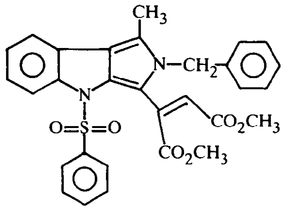
Abstract

The title molecule, $C_{30}H_{26}N_2O_6S$, consists of a methylpyrrolo[3,4-*b*]indole ring with a phenylsulfonyl, a phenyl and a dimethyl maleate group as exocyclic substituents. The pyrrolo[3,4-*b*]indole ring system is planar. The S atom has distorted tetrahedral geometry. The dihedral angle between the pyrrolo[3,4-*b*]indole and the phenylsulfonyl group is 65.2 (4) and it is 75.69 (1)° with the phenyl group. The sum of angles at the indole-N atom is 341.7°, while the second pyrrole ring N atom adopts sp^2 hybridization. The packing is stabilized by weak C—H···O hydrogen bonds.

Comment

Indole alkaloids are known for their interesting chemical, medicinal and physiological activities. A variety of [*b*]annellated indoles are of biological interest as antitumour-active substances (Schollmeyer *et al.*, 1995). The spiro-indole derivatives exhibit antibacterial and antifungal properties (Sehgal *et al.*, 1994). These types of compounds are also used as antimicrobial and anti-inflammatory agents (El-Sayed *et al.*, 1986; Rodriguez *et al.*, 1985). The sulfonamide-containing drugs behave as diuretics, and sulthiame, as a carbonic anhydrase inhibitor (Crawford & Kennedy, 1959; Camerman & Camerman, 1975; Tanimukai *et al.*, 1976). The sulfonamides inhibit growing bacterial organisms and are also useful for treating urinary and gastrointestinal infections. The X-ray structural study of the title compound, (I), has been carried out in continuation of our studies (Govindasamy *et al.*, 1997*a*,*b*, 1998*a*,*b*; Subramanian *et al.*, 1997).



(I)

A *ZORTEP* (Zsolnai, 1997) plot of the molecule with atomic numbering scheme is shown in Fig. 1. The S atom has a distorted tetrahedral geometry. The N atoms, N1 and N2, occupy axial orientations with respect to their substituents. The C—N distances in ring *B* [N1—C1 = 1.465 (2), N1—C10 = 1.451 (2) Å] are significantly larger than in ring *C* [N2—C8 = 1.376 (2), N2—C9 = 1.411 (2) Å], in agreement with the electron-withdrawing character of the phenylsulfonyl group (Govindasamy *et al.*, 1998*b*; Preut *et al.*, 1988). The bond distances S—O, S—C and S—N are 1.424 (2), 1.755 (2) and 1.678 (2) Å, respectively, and are comparable with reported values (Govindasamy *et al.*, 1998*a*). The C18—C22 bond length is slightly larger than expected. The conformation of the molecule may be described by the torsion angles N1—S1—C11—C16 and N1—S1—C11—C12, which are −89.98 (19) and 90.8 (2)°, respectively, and lie within the range $|\varepsilon_1|$ = 60–90° (Cotton & Stokely, 1970).

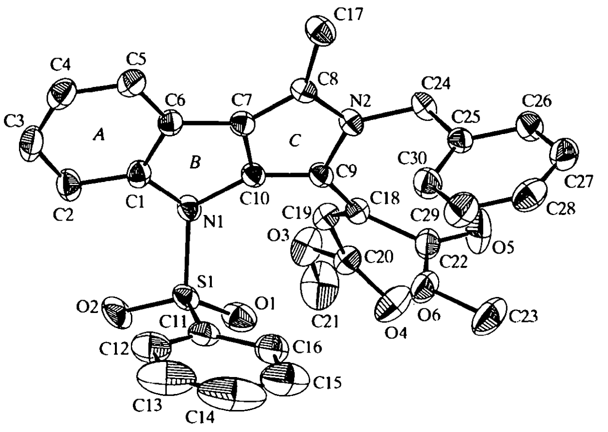


Fig. 1. Molecular structure showing 30% probability displacement ellipsoids with the atom-numbering scheme.

The three rings of the fused heteroarene are nearly planar. The planarity of the tricyclic hetarene is an essential feature for delocalization; distortion should be favourable for localized five- and six-membered π systems. The sum of the angles at N1 is 341.7°, indicating a significant pyramidal character.

The position of the methyl carbon C17 atom at the C8 atom of the second pyrrol ring, *C*, does not deviate significantly from the least-squares plane through the pyrrol ring *C*. Thus the N2 atom may be considered sp^2 hybridized. The sulfonyl group is oriented in such a way as to have both the N atom lone pair and the *p* orbital at the sulfur-bearing aromatic-C atom nearly coplanar, the plane bisecting the O—S—O angle.

In the indole ring, the angles at C2 and C5 are contracted to 117.1 (2) and 119.3 (2)°, respectively, while those at C3 and C6 are expanded to 122.1 (2) and 119.9 (2)°, respectively. This would appear to be a real effect caused by the fusion of a smaller pyrrole ring to the six-membered phenyl ring and the strain is taken up by angular distortion rather than by bond-length distortions. The angles C11—S1—N1—C10 and C11—S1—N1—C1 are 64.2 (2) and −64.1 (2)°, respectively, and lie within the range reported in ethyl 4,6-dimethoxy-9-phenylsulfonylcarbazole-3-carboxylate (Govindasamy *et al.*, 1997*b*). The dihedral angle between the pyrrolo[3,4-*b*]indole moiety and phenylsulfonyl group is 65.2 (4) and with the phenyl group it is 75.69 (1)°.

In addition to van der Waals forces, the packing of the molecules in the unit cell is governed by C—H···O hydrogen bonds.

Experimental

The synthesis of the title compound is reported elsewhere (Jeevanandam & Srinivasan, 1995). Single crystals were obtained by slow evaporation from methanol/chloroform mixture.

Crystal data

$C_{30}H_{26}N_2O_6S$
$M_r = 542.59$
Monoclinic
$P2_1/c$
$a = 10.2215\ (2)$ Å
$b = 20.8692\ (4)$ Å
$c = 12.8700\ (1)$ Å
$\beta = 98.130\ (1)°$
$V = 2717.77\ (8)$ Å^3
$Z = 4$
$D_x = 1.326$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 39 reflections
$\theta = 5.35–12.41°$
$\mu = 0.166$ mm^{-1}
$T = 293\ (2)$ K
Block
0.52 × 0.46 × 0.42 mm
Yellow

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: empirical (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.758$, $T_{\max} = 0.968$
17 476 measured reflections
6214 independent reflections

4646 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 27.50°$
$h = -13 \rightarrow 13$
$k = -17 \rightarrow 27$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.176$
$S = 1.129$
6214 reflections
355 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.11P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.218$ e Å^{-3}
$\Delta\rho_{\min} = -0.365$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

S1—O1	1.423 (2)	N2—C24	1.455 (2)
S1—O2	1.424 (2)	O3—C20	1.330 (2)
S1—N1	1.678 (2)	O3—C21	1.438 (3)
S1—C11	1.755 (2)	O4—C20	1.202 (2)
N1—C10	1.451 (2)	O5—C22	1.191 (3)
N1—C1	1.465 (2)	O6—C22	1.335 (3)
N2—C8	1.376 (2)	O6—C23	1.458 (3)
N2—C9	1.411 (2)	C18—C22	1.516 (3)
O1—S1—O2	119.3 (1)	C22—O6—C23	116.5 (2)
O1—S1—N1	107.11 (9)	C2—C1—N1	128.1 (2)
O2—S1—N1	106.08 (9)	C6—C1—N1	110.6 (2)
O1—S1—C11	108.7 (1)	N2—C8—C7	106.5 (2)
O2—S1—C11	108.77 (10)	N2—C8—C17	123.2 (2)
N1—S1—C11	106.16 (9)	C7—C8—C17	130.3 (2)
C10—N1—C1	104.3 (1)	C10—C9—N2	104.2 (2)
C8—N2—C9	111.3 (2)	N2—C9—C18	123.4 (2)
C8—N2—C24	122.8 (2)	C19—C18—C22	122.6 (2)
C9—N2—C24	125.7 (2)	C18—C19—C20	127.7 (2)
C20—O3—C21	117.0 (2)		
C11—S1—N1—C10	64.2 (2)	C11—S1—N1—C1	−64.1 (2)

Data collection: *SMART* (Siemens, 1996*a*). Cell refinement: *SAINT* (Siemens, 1996*b*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*b*). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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