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[2-(3,4-Methylenedioxyphenyl)-1-(phenylsulfonyl)vinyl]-3-(phenylthio)indole

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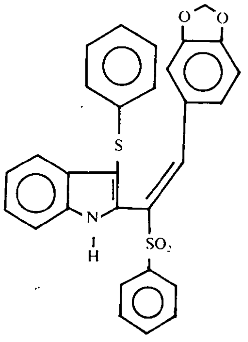
Abstract

The dioxole ring of the title compound, $C_{29}H_{21}NO_4S_2$, is inclined at an angle of 67.9 (2)° to the indole ring system. The phenyl rings of the phenylsulfonyl and phenylthio substituents are almost perpendicular to each other [73.3 (2)°], while the phenylthio and the phenyl ring of the methylenedioxyphenyl ring system are almost parallel to each other [8.2 (2)°]. The indole ring system is slightly folded along the central C—C bond (2.1°). All the rings are quite planar. The molecules are linked by N—H···O-type hydrogen bonds.

Comment

2-Vinylindole and its various substituted products have long been known for their interesting chemical and biological activities. Compounds of this class are reported to exhibit antimicrobial (El-Sayed, Barnhart, Ammon & Wassel, 1986) and anti-inflammatory activities (Rodriguez, Temprano, Estebancalderon, Martinez-Ripoll & Garciablanco, 1985). The present study constitutes part of a series of studies on the structure and conformation of substituted indoles, which has been undertaken in order to correlate chemical structure and biological activity.

The bond parameters of the title compound (I) have expected values. The bond angles in all the six-membered rings have an average value of 120.01 (1)° but there are significant deviations among the individual values. In the indole ring system, the angles at C(7) and C(4) are contracted to 117.7 (4) and 118.0 (4)°, respectively, while those at C(8) and C(5) are expanded to 121.9 (4) and 121.2 (4)°, respectively. This would appear to be a real effect caused by the fusion of a smaller pyrrole ring to the six-membered phenyl ring, the strain being taken up by angular distortion rather than bond-length distortion. This effect has been observed by Allen & Trotter (1970). The same effect is also observed in the methylenedioxyphenyl part of the molecule [angles at C(2*b*) and C(5*b*) are contracted to 117.0 (4) and 116.9 (4)°, respectively, whereas those at C(3*b*) and C(6*b*) are expanded to 122.9 (4) and 122.4 (4)°, respectively] because of the fusion of the phenyl ring with the dioxole ring. The sum of the angles around C(1′) is 359.6°. It has been found that the indole system is nearly planar ($\chi^2 = 12.6$°) (Chakraborty & Talapatra, 1986) with a maximum deviation for C(9) (0.025 Å) from the mean plane containing atoms N(1) and C(2)–C(9). The dihedral angles between the indole ring system and the mean planes of the phenylthio phenyl ring, the methylenedioxy phenyl



(I)

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system and the phenylsulfonyl phenyl ring are 65.64 (13), 67.98 (9) and 138.03 (13)°, respectively. The phenylthio and phenylsulfonyl phenyl groups are almost perpendicular to each other, the angle between them being 73.29 (16)°. It was also observed that the methylenedioxyphenyl ring system and the phenylthio phenyl ring are almost parallel, the angle between them being 8.2 (2)°.

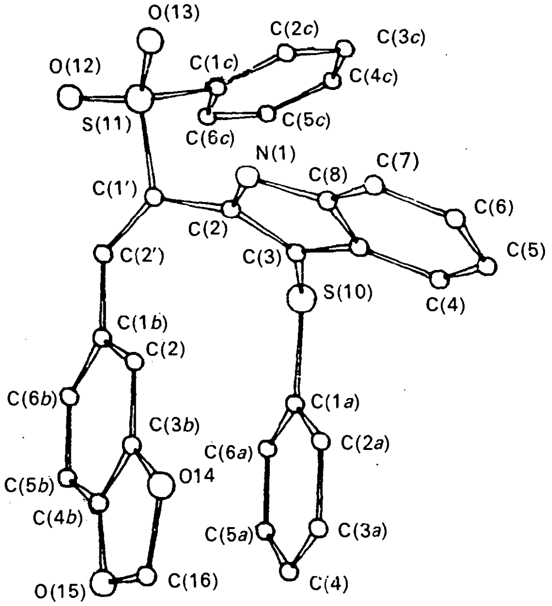

Fig. 1. Atomic numbering scheme of the title molecule.

Experimental

Crystal data

$C_{29}H_{21}NO_4S_2$
$M_r = 511.6$
Orthorhombic
Pbcn
$a = 15.800\ (2)$ Å
$b = 14.345\ (2)$ Å
$c = 21.424\ (1)$ Å
$V = 4855.8\ (9)$ Å^3
$Z = 8$
$D_x = 1.40$ Mg m^{-3}
$D_m = 1.321$ Mg m^{-3}
D_m measured by flotation

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 20 reflections
$\theta = 15$-$25°$
$\mu = 2.25$ mm^{-1}
$T = 293$ K
Rectangular blocks
0.40 × 0.40 × 0.35 mm
Pale yellow

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
$T_{\min} = 0.817$, $T_{\max} = 0.925$
3388 measured reflections
3195 independent reflections
2896 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int} = 0.009$
$\theta_{\max} = 60°$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 24$
2 standard reflections monitored every 100 reflections
intensity variation: <2%

Refinement

Refinement on F
$R = 0.054$
$wR = 0.06$
$S = 1.43$
2896 reflections
409 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F) + 0.028F^2]$
$(\Delta/\sigma)_{\max} = 0.089$
$\Delta\rho_{\max} = 0.26$ e Å^{-3}
$\Delta\rho_{\min} = -0.46$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
N(1)	0.5995 (2)	0.3726 (2)	0.4990 (1)	3.64 (8)
C(2)	0.6016 (2)	0.3393 (2)	0.5592 (2)	3.31 (9)
C(3)	0.6344 (2)	0.2510 (2)	0.5596 (2)	3.35 (9)
C(4)	0.6879 (3)	0.1483 (3)	0.4667 (2)	4.48 (11)
C(5)	0.7013 (3)	0.1513 (3)	0.4033 (2)	5.36 (13)
C(6)	0.6803 (3)	0.2310 (4)	0.3681 (2)	5.33 (13)
C(7)	0.6464 (3)	0.3083 (3)	0.3953 (2)	4.83 (12)
C(8)	0.6321 (2)	0.3058 (3)	0.4597 (2)	3.72 (9)
C(9)	0.6526 (2)	0.2278 (3)	0.4957 (2)	3.49 (10)
S(10)	0.6466 (1)	0.1812 (1)	0.6257 (1)	4.19 (3)
S(11)	0.4613 (1)	0.4089 (1)	0.6182 (1)	4.20 (3)
O(12)	0.4405 (2)	0.4594 (2)	0.6741 (2)	5.90 (11)
O(13)	0.4326 (2)	0.4446 (2)	0.5588 (2)	5.85 (10)
O(14)	0.9042 (2)	0.4333 (3)	0.5647 (2)	7.75 (13)
O(15)	0.9724 (2)	0.4290 (3)	0.6602 (2)	7.36 (13)
C(16)	0.9849 (3)	0.4275 (6)	0.5946 (4)	9.0 (3)
C(1')	0.5724 (3)	0.3952 (3)	0.6132 (2)	3.70 (10)
C(2')	0.6198 (3)	0.4261 (3)	0.6597 (2)	4.03 (10)
C(1*a*)	0.7585 (3)	0.1812 (3)	0.6394 (2)	3.81 (10)
C(2*a*)	0.8172 (3)	0.1914 (5)	0.5942 (2)	6.52 (18)
C(3*a*)	0.9037 (3)	0.1845 (5)	0.6084 (2)	6.91 (17)
C(4*a*)	0.9289 (3)	0.1692 (4)	0.6680 (3)	5.45 (16)
C(5*a*)	0.8691 (4)	0.1582 (5)	0.7133 (2)	6.67 (17)
C(6*a*)	0.7842 (3)	0.1648 (4)	0.6997 (2)	5.83 (14)
C(1*b*)	0.7124 (3)	0.4273 (3)	0.6616 (2)	3.98 (10)
C(2*b*)	0.7606 (3)	0.4313 (3)	0.6061 (2)	4.60 (12)
C(3*b*)	0.8461 (3)	0.4304 (4)	0.6123 (2)	4.96 (13)
C(4*b*)	0.8858 (3)	0.4272 (3)	0.6690 (2)	5.02 (13)
C(5*b*)	0.8404 (3)	0.4251 (4)	0.7240 (2)	5.74 (14)
C(6*b*)	0.7539 (3)	0.4258 (3)	0.7191 (2)	4.92 (13)
C(1*c*)	0.4232 (2)	0.2945 (3)	0.6251 (2)	3.76 (10)
C(2*c*)	0.4008 (3)	0.2449 (4)	0.5732 (2)	5.28 (14)
C(3*c*)	0.3738 (4)	0.1513 (4)	0.5813 (3)	7.00 (18)
C(4*c*)	0.3741 (4)	0.1121 (4)	0.6405 (3)	6.88 (17)
C(5*c*)	0.3981 (3)	0.1622 (4)	0.6907 (3)	5.91 (15)
C(6*c*)	0.4219 (3)	0.2553 (3)	0.6844 (2)	4.83 (13)

Table 2. *Selected geometric parameters* (Å, °)

N(1)—C(2)	1.376 (5)	C(1')—C(2')	1.323 (6)
N(1)—C(8)	1.376 (5)	C(2')—C(1*b*)	1.464 (7)
C(2)—C(3)	1.369 (4)	C(1*a*)—C(2*a*)	1.349 (6)
C(2)—C(1')	1.481 (6)	C(1*a*)—C(6*a*)	1.375 (6)
C(3)—C(9)	1.438 (6)	C(2*a*)—C(3*a*)	1.404 (7)
C(3)—S(10)	1.745 (4)	C(3*a*)—C(4*a*)	1.355 (8)
C(4)—C(5)	1.375 (6)	C(4*a*)—C(5*a*)	1.364 (8)
C(4)—C(9)	1.413 (6)	C(5*a*)—C(6*a*)	1.376 (8)
C(5)—C(6)	1.409 (7)	C(1*b*)—C(2*b*)	1.413 (3)
C(6)—C(7)	1.362 (7)	C(1*b*)—C(6*b*)	1.396 (6)
C(7)—C(8)	1.399 (6)	C(2*b*)—C(3*b*)	1.358 (7)
C(8)—C(9)	1.397 (6)	C(3*b*)—C(4*b*)	1.368 (6)
S(10)—C(1*a*)	1.792 (5)	C(4*b*)—C(5*b*)	1.380 (6)
S(11)—O(12)	1.438 (4)	C(5*b*)—C(6*b*)	1.371 (7)
S(11)—O(13)	1.445 (5)	C(1*c*)—C(2*c*)	1.367 (6)
S(11)—C(1')	1.770 (5)	C(1*c*)—C(6*c*)	1.390 (6)
S(11)—C(1*c*)	1.754 (4)	C(2*c*)—C(3*c*)	1.420 (8)
O(14)—C(16)	1.429 (7)	C(3*c*)—C(4*c*)	1.387 (9)

O(14)—C(3*b*)	1.373 (6)	C(4*c*)—C(5*c*)	1.348 (9)
O(15)—C(16)	1.420 (10)	C(5*c*)—C(6*c*)	1.394 (7)
O(15)—C(4*b*)	1.381 (6)		
C(2)—N(1)—C(8)	108.8 (3)	C(1′)—C(2′)—C(1*b*)	126.2 (4)
N(1)—C(2)—C(1′)	122.5 (3)	S(10)—C(1*a*)—C(6*a*)	116.5 (4)
N(1)—C(2)—C(3)	109.7 (4)	S(10)—C(1*a*)—C(2*a*)	124.1 (4)
C(3)—C(2)—C(1′)	127.9 (3)	C(2*a*)—C(1*a*)—C(6*a*)	119.4 (4)
C(2)—C(3)—S(10)	125.3 (3)	C(1*a*)—C(2*a*)—C(3*a*)	120.4 (4)
C(2)—C(3)—C(9)	106.5 (3)	C(2*a*)—C(3*a*)—C(4*a*)	120.1 (5)
C(9)—C(3)—S(10)	128.2 (3)	C(3*a*)—C(4*a*)—C(5*a*)	119.1 (5)
C(5)—C(4)—C(9)	118.0 (4)	C(4*a*)—C(5*a*)—C(6*a*)	121.1 (4)
C(4)—C(5)—C(6)	121.2 (4)	C(1*a*)—C(6*a*)—C(5*a*)	119.9 (4)
C(5)—C(6)—C(7)	121.6 (4)	C(2′)—C(1*b*)—C(6*b*)	119.6 (4)
C(6)—C(7)—C(8)	117.7 (4)	C(2′)—C(1*b*)—C(2*b*)	121.1 (4)
N(1)—C(8)—C(7)	130.3 (4)	C(2*b*)—C(1*b*)—C(6*b*)	119.4 (4)
C(7)—C(8)—C(9)	121.9 (4)	C(1*b*)—C(2*b*)—C(3*b*)	117.0 (4)
N(1)—C(8)—C(9)	107.9 (3)	O(14)—C(3*b*)—C(2*b*)	126.3 (4)
C(4)—C(9)—C(8)	119.7 (4)	C(2*b*)—C(3*b*)—C(4*b*)	122.9 (4)
C(3)—C(9)—C(8)	107.1 (3)	O(14)—C(3*b*)—C(4*b*)	110.7 (4)
C(3)—C(9)—C(4)	133.2 (4)	O(15)—C(4*b*)—C(3*b*)	109.4 (4)
C(3)—S(10)—C(1*a*)	104.0 (2)	C(3*b*)—C(4*b*)—C(5*b*)	121.4 (4)
C(1′)—S(11)—C(1*c*)	104.0 (2)	O(15)—C(4*b*)—C(5*b*)	129.2 (4)
O(13)—S(11)—C(1*c*)	107.3 (2)	C(4*b*)—C(5*b*)—C(6*b*)	116.9 (4)
O(13)—S(11)—C(1′)	107.3 (2)	C(1*b*)—C(6*b*)—C(5*b*)	122.4 (4)
O(12)—S(11)—C(1*c*)	108.8 (2)	S(11)—C(1*c*)—C(6*c*)	117.4 (3)
O(12)—S(11)—C(1′)	109.5 (2)	S(11)—C(1*c*)—C(2*c*)	120.5 (3)
O(12)—S(11)—O(13)	118.9 (2)	C(2*c*)—C(1*c*)—C(6*c*)	122.0 (4)
C(16)—O(14)—C(3*b*)	105.2 (4)	C(1*c*)—C(2*c*)—C(3*c*)	118.1 (4)
C(16)—O(15)—C(4*b*)	105.8 (4)	C(2*c*)—C(3*c*)—C(4*c*)	119.6 (5)
O(14)—C(16)—O(15)	108.6 (4)	C(3*c*)—C(4*c*)—C(5*c*)	121.0 (6)
C(2)—C(1′)—S(11)	114.6 (3)	C(4*c*)—C(5*c*)—C(6*c*)	120.6 (6)
S(11)—C(1′)—C(2′)	118.6 (4)	C(1*c*)—C(6*c*)—C(5*c*)	118.7 (4)
C(2)—C(1′)—C(2′)	126.4 (4)		

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(1)—H(1)···O(13)	0.88	2.78	3.108 (4)	103
C(2′)—H(2′)···O(12)	0.92	2.51	2.889 (6)	105
C(2*c*)—H(2*c*)···O(13)	0.95	2.55	2.925 (6)	103
C(6*c*)—H(6*c*)···O(12)	1.01	2.55	2.951 (5)	103
N(1)—H(1)···O(13^{i})	0.88	2.10	2.944 (4)	160

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

Cell refinement and data reduction: *SDP* (Frenz, 1978). Program used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program used to refine structure: *SHELX*76 (Sheldrick, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983) and *PLUTO* (Motherwell, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances involving H atoms, least-squares-planes data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71541 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1061]

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2-[2-(4-Methoxyphenyl)-1-(phenylsulfonyl)-vinyl]-3-(phenylthio)indole

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(*Received* 18 *June* 1992; *accepted* 15 *February* 1993)

Abstract

In the title compound, $C_{29}H_{23}NO_3S_2$, the phenylthio, phenylsulfonyl and anisole rings are inclined at 76.4 (1), 60.0 (1) and 102.6 (1)°, respectively, to the indole ring. The phenylthio and phenylsulfonyl rings are nearly parallel to each other, the dihedral angle between them being 17.6 (1)°.

Comment

The crystal and molecular structure of the title compound (I) was investigated to determine the stereochemistry of the substituents with respect to the indole ring system. Compounds of this class are reported to exhibit antimicrobial (El-Sayed, Barnhart, Ammon & Wassel, 1986) and anti-inflammatory activity (Rodriguez, Temprano, Estebancalderon, Martinez-Ripoll & Garciablanco, 1985).

The bond angles of all the six-membered rings have an average value of 120.0° but there are significant differences between individual values. In the indole ring system, the angles at C(7) and C(4) are

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