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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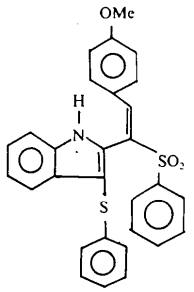
Abstract

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



(I)

contracted to 117.0 (2) and 118.3 (2)°, respectively, while those at C(8) and C(5) are expanded to 123.0 (2) and 122.3 (4)°, respectively. This would appear to be a real effect caused by the fusion of a smaller pyrrole ring to a phenyl ring; the strain is taken up by angular rather than bond-length distortion. Similar effects have been observed previously (Allen & Trotter, 1970). The methoxy group at 2C(4) is twisted slightly out of the plane of the benzene ring [2C(3)—2C(4)—O(14)—C(15) 10.2 (3)°]. There is possibly an intramolecular hydrogen bond between N(1) and O(13) (see Table 2). The proton on atom C(2)′ may be involved in an intramolecular interaction with O(12), and 3C(6) makes a close contact with O(14) from a neighbouring molecule. Intermolecular packing involves van der Waals interactions only.

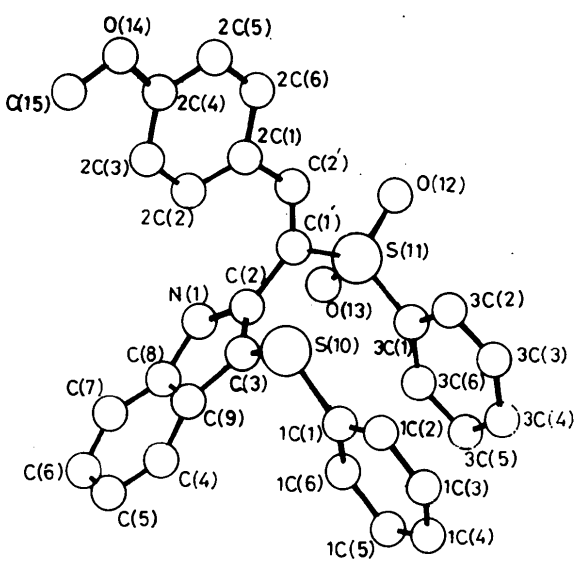


Fig. 1. One molecule of the title compound; H atoms are omitted for clarity.

Experimental

Crystal data

$C_{29}H_{23}NO_3S_2$
M_r = 497.6
Monoclinic
$C2/c$
a = 24.414 (2) Å
b = 11.587 (2) Å
c = 21.108 (1) Å
β = 123.33 (3)°
V = 4989.0 Å^3
Z = 8
D_x = 1.325 Mg m^{-3}
D_m = 1.322 Mg m^{-3}
D_m measured by flotation
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 20 reflections
θ = 20–30°
μ = 2.14 mm^{-1}
T = 293 K
Rectangular blocks
0.4 × 0.3 × 0.3 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
T_{min} = 0.653, T_{max} = 0.721
3564 measured reflections
3129 independent reflections
3129 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.0110
θ_{max} = 60°
h = −27 → 23
k = 0 → 12
l = 0 → 23
2 standard reflections monitored every 100 reflections
intensity variation: <2%

Refinement

Refinement on F
R = 0.039
wR = 0.048
S = 1.605
3129 reflections
408 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.014449F^2]$
$(\Delta/\sigma)_{max}$ = 0.008
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

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

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
N(1)	0.0686 (1)	0.1478 (1)	1.0946 (1)	0.0408 (9)
C(2)	0.1120 (1)	0.1946 (1)	1.0796 (1)	0.0359 (10)
C(3)	0.1707 (1)	0.2088 (2)	1.1453 (1)	0.0416 (11)
C(4)	0.2052 (1)	0.1581 (2)	1.2827 (1)	0.0586 (14)
C(5)	0.1812 (2)	0.1129 (3)	1.3229 (1)	0.0808 (19)
C(6)	0.1166 (2)	0.0755 (3)	1.2879 (1)	0.085 (2)
C(7)	0.0744 (1)	0.0833 (2)	1.2118 (1)	0.0706 (16)
C(8)	0.0988 (1)	0.1301 (2)	1.1709 (1)	0.0458 (11)
C(9)	0.1631 (1)	0.1680 (2)	1.2050 (1)	0.0439 (11)
S(10)	0.2375 (2)	0.2799 (1)	1.1545 (2)	0.057 (4)
S(11)	0.0814 (1)	0.0991 (2)	0.9460 (1)	0.0395 (3)
O(12)	0.0589 (1)	0.1366 (1)	0.8711 (1)	0.0513 (8)
O(13)	0.0429 (1)	0.0169 (1)	0.9569 (1)	0.0051 (8)
O(14)	0.0691 (1)	0.7837 (1)	1.0439 (1)	0.0542 (9)
C(15)	0.0936 (1)	0.8203 (2)	1.1193 (1)	0.0633 (15)
C(1′)	0.0925 (1)	0.2219 (2)	1.0022 (1)	0.0367 (10)
C(2′)	0.0808 (1)	0.3243 (2)	0.9679 (1)	0.0420 (11)
1C (1)	0.2986 (1)	0.1731 (3)	1.1862 (1)	0.0510 (14)
1C (2)	0.3578 (1)	0.2116 (3)	1.1988 (2)	0.085 (2)
1C (3)	0.4075 (2)	0.1348 (6)	1.2223 (2)	0.122 (3)
1C (4)	0.4012 (2)	0.0219 (6)	1.2354 (2)	0.115 (3)
1C (5)	0.3431 (2)	−0.0193 (3)	1.2240 (1)	0.083 (2)
1C (6)	0.2910 (1)	0.0582 (3)	1.1986 (1)	0.0624 (16)
2C (1)	0.0804 (1)	0.4414 (2)	0.9938 (1)	0.0405 (11)

2C (2)	0.0953 (1)	0.4707 (2)	1.0653 (1)	0.0480 (11)
2C (3)	0.0930 (1)	0.5837 (2)	1.0842 (1)	0.0503 (12)
2C (4)	0.0744 (1)	0.6704 (2)	1.0312 (1)	0.0671 (11)
2C (5)	0.0593 (1)	0.6432 (2)	0.9602 (1)	0.0535 (13)
2C (6)	0.0623 (1)	0.5304 (2)	0.9416 (1)	0.0473 (12)
3C (1)	0.1595 (1)	0.0385 (2)	0.9848 (1)	0.0417 (11)
3C (2)	0.2065 (1)	0.0994 (2)	0.9807 (1)	0.0549 (13)
3C (3)	0.2662 (1)	0.0488 (2)	1.0078 (1)	0.0674 (16)
3C (4)	0.2780 (1)	−0.0612 (3)	1.0365 (1)	0.0638 (16)
3C (5)	0.2313 (1)	−0.1216 (2)	1.0393 (1)	0.0654 (13)
3C (6)	0.1708 (1)	−0.0713 (2)	1.0137 (1)	0.0546 (13)

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

N(1)—C(2)	1.373 (4)	C(2′)—2C(1)	1.465 (3)
N(1)—C(8)	1.369 (3)	1C(1)—1C(2)	1.391 (4)
C(2)—C(3)	1.349 (2)	1C(1)—1C(6)	1.389 (5)
C(2)—C(1′)	1.463 (3)	1C(2)—1C(3)	1.360 (6)
C(3)—C(9)	1.450 (4)	1C(3)—1C(4)	1.363 (10)
C(3)—S(10)	1.740 (5)	1C(4)—1C(5)	1.387 (7)
C(4)—C(5)	1.373 (5)	1C(5)—1C(6)	1.401 (5)
C(4)—C(9)	1.381 (2)	2C(1)—2C(2)	1.386 (3)
C(5)—C(6)	1.393 (6)	2C(1)—2C(6)	1.392 (3)
C(6)—C(7)	1.353 (2)	2C(2)—2C(3)	1.379 (3)
C(7)—C(8)	1.400 (4)	2C(3)—2C(4)	1.382 (3)
C(8)—C(9)	1.391 (3)	2C(4)—2C(5)	1.369 (3)
S(10)—1C(1)	1.763 (4)	2C(5)—2C(6)	1.378 (3)
S(11)—O(12)	1.427 (3)	3C(1)—3C(2)	1.390 (4)
S(11)—O(13)	1.443 (3)	3C(1)—3C(6)	1.372 (3)
S(11)—C(1′)	1.775 (3)	3C(2)—3C(3)	1.371 (3)
S(11)—3C(1)	1.756 (3)	3C(3)—3C(4)	1.372 (4)
O(14)—C(15)	1.423 (3)	3C(4)—3C(5)	1.366 (4)
O(14)—2C(4)	1.360 (3)	3C(5)—3C(6)	1.392 (3)
C(1′)—C(2′)	1.337 (3)		
C(2)—N(1)—C(8)	110.2 (2)	1C(3)—1C(4)—1C(5)	120.9 (5)
N(1)—C(2)—C(3)	109.2 (2)	1C(4)—1C(5)—1C(6)	118.2 (4)
C(2)—C(3)—C(9)	106.4 (2)	1C(5)—1C(6)—1C(1)	120.0 (3)
C(5)—C(4)—C(9)	118.3 (2)	2C(1)—2C(2)—2C(3)	121.3 (2)
C(4)—C(5)—C(6)	122.3 (4)	2C(2)—2C(3)—2C(4)	120.3 (2)
C(5)—C(6)—C(7)	120.7 (2)	2C(3)—2C(4)—2C(5)	119.3 (2)
C(6)—C(7)—C(8)	117.0 (2)	2C(4)—2C(5)—2C(6)	120.4 (2)
N(1)—C(8)—C(7)	130.3 (2)	2C(5)—2C(6)—2C(1)	121.4 (2)
C(7)—C(8)—C(9)	123.0 (2)	2C(6)—2C(1)—2C(2)	117.4 (2)
N(1)—C(8)—C(9)	106.7 (2)	3C(2)—3C(3)—3C(4)	119.9 (3)
C(4)—C(9)—C(8)	118.7 (2)	3C(3)—3C(4)—3C(5)	121.1 (3)
C(3)—C(9)—C(8)	107.5 (2)	3C(4)—3C(5)—3C(6)	120.1 (3)
C(3)—C(9)—C(4)	133.8 (2)	3C(1)—3C(6)—3C(5)	118.2 (2)
1C(2)—1C(1)—1C(6)	120.2 (3)	3C(6)—3C(1)—3C(2)	121.8 (2)
1C(1)—1C(2)—1C(3)	119.0 (4)	3C(1)—3C(2)—3C(3)	118.8 (3)
1C(2)—1C(3)—1C(4)	121.6 (5)		
N(1)—H(1)···O(13)	3.023 (3)	3C(6)—H3(6)···O(14^{i})	3.337 (4)
C(2′)—H(2′)···O(12)	2.827 (3)		

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

Cell refinement and data reduction: *SDP* (Frenz, 1978). Program used to solve structure: *SHELXS86* (Sheldrick, 1985). Program used to refine structure: *SHELX76* (Sheldrick, 1976). Software used to prepare material for publication: *PARST* (Nardell, 1983) and *PLUTO* (Motherwell, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71182 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1018]

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Acta Cryst. (1994). C**50**, 791–794

2-Benzoyl-3-(4-methoxyphenyl)-4-methyl-1-phenyl-2,3-dihydro-1*H*-pyrazolo[4,3-*c*]-[1,2]benzothiazine 5,5-Dioxide

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Abstract

Two molecules of the title compound, $C_{30}H_{25}N_3O_4S$, crystallize in the asymmetric unit. The phenyl, benzoyl and methoxyphenyl substituents point alternately up and down with respect to the dihydropyrazolo cycle (in agreement with steric effects). The conformation of the molecule is such that the thiazine cycle has an approximate plane of symmetry passing through the S atom (and the two O atoms), whereas the dihydropyrazolo cycle has a twofold axis passing between the two N atoms.

Comment

The reaction of diphenylnitrilimine (1) with 3-arylidene-2*H*-3,4-dihydro-1,2-benzothiazin-4-one