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2,3-Bis(dibromomethyl)-1-(phenylsulfonyl)-1*H*-indole

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Key indicators

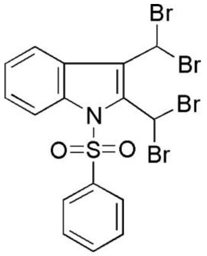
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.023
wR factor = 0.042
Data-to-parameter ratio = 24.7

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

In the structure of the title compound, $C_{16}H_{11}Br_4NO_2S$, the two dibromomethyl groups are rotated in a such a way as to minimize their mutual interactions. The dihedral angle between the phenylsulfonyl group and the mean plane of the indole ring is 86.1 (7)°. The non-fused pyrrole C—C bond length is 1.362 (3)Å, the S—N distance is 1.685 (4)Å, and the average S=O distance is 1.424 (3)Å.

Comment

Pursuant to the generation of novel indole-2,3-quinodimethanes, which are very useful in cycloaddition reactions (Saroja & Srinivasan, 1984; Magnus *et al.*, 1984; Haber & Pindur, 1991; Pindur & Erfanian-Abdoust, 1989), we have synthesized the title compound, (I), and determined its crystal structure (Fig. 1). The observed parameters (Table 1) are comparable to the reported values for other *N*-(phenylsulfonyl)indoles (Beddoes *et al.*, 1986; Schollmeyer *et al.*, 1995; Yokum & Fronczek, 1997; Govindasamy *et al.*, 1998; Sankaranarayanan *et al.*, 2000; Sonar *et al.*, 2004; Palani *et al.*, 2006*a*,*b*).



(I)

The sum of the angles around the indole N atom is 357.7°, indicating nearly ideal sp^2 hybridization. The four C—Br bond lengths average 1.947 (3) Å, comparable to the distance of 1.959 (3) Å reported for 3-bromomethyl-2-cyano-1-(phenylsulfonyl)indole (Palani *et al.*, 2006*a*). Both Br—CH—Br groups have approximate mirror symmetry across the indole ring plane. The C16—C2—C3—C17 torsion angle is −0.9 (4)°, revealing no serious out-of-plane distortion of the two dibromomethyl carbons. The C2=C3 indole double-bond length is 1.373 (3) Å, which is significantly longer than the indole double-bond lengths of 1.349 (11) Å in 2,3-diphenyl-1-(phenylsulfonyl)indole (Liu *et al.*, 2007) and 1.341 (3) Å in 2,3-dimethyl-1-(phenylsulfonyl)indole (Palani *et al.*, 2006*b*).

Experimental

A mixture of 2,3-dimethyl-1-(phenylsulfonyl)-1*H*-indole (0.220 g, 0.772 mmol), *N*-bromosuccinimide (0.557 g, 3.13 mmol) and benzoyl

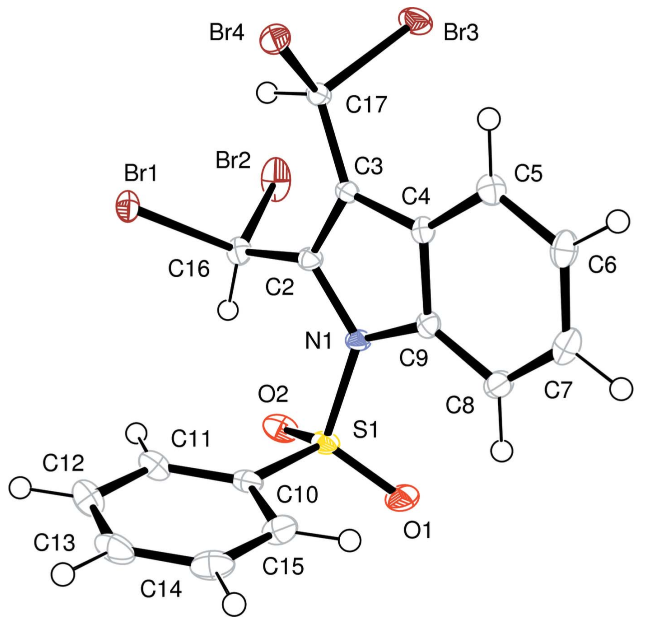


Figure 1
The molecular structure of (I) showing 50% probability displacement ellipsoids. H atoms are drawn as spheres of arbitrary radius.

peroxide (0.0585 g, 0.423 mmol) was stirred in carbon tetrachloride (25 ml). The yellow suspension was refluxed for 6 h, after which it was allowed to cool to room temperature. It was poured into distilled water (50 ml). The organic layer was extracted with hexanes (2 × 25 ml, then 1 × 50 ml), washed with distilled water (2 × 50 ml), and dried with anhydrous magnesium sulfate. The solvent was evaporated *in vacuo* to yield a yellow solid (0.383 g, 0.637 mmol, 82%; m.p. 434–435 K). HRMS calculated for $C_{16}H_{11}Br_4NO_2S$: 596.7244; found: 596.7232. Crystals suitable for the X-ray study were grown from diethyl ether–hexane (1:1).

Crystal data

$C_{16}H_{11}Br_4NO_2S$
$M_r = 600.95$
Monoclinic, $P2_1/c$
$a = 9.5701\ (3)$ Å
$b = 17.3004\ (6)$ Å
$c = 10.9621\ (4)$ Å
$\beta = 91.5878\ (15)^\circ$
$V = 1814.26\ (11)$ Å^3
$Z = 4$
$D_x = 2.200$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 9.00$ mm^{-1}
$T = 120$ K
Prism, colorless
0.42 × 0.39 × 0.32 mm

Data collection

Bruker-Nonius Kappa APEX2 diffractometer
φ and ω scans
Absorption correction: numerical (de Meulenaer & Tompa, 1965) $T_{\min} = 0.03$, $T_{\max} = 0.06$
24244 measured reflections
5383 independent reflections
3962 reflections with $I > 2.0\sigma(I)$
$R_{\rm int} = 0.047$
$\theta_{\max} = 30.3^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.042$
$S = 1.00$
5383 reflections
218 parameters
H-atom parameters constrained
$w = [1 - (F_o - F_c)^2/36\sigma^2(F)]^2/[7.56T_0(x) + 7.82T_1(x) + 2.08T_2(x)]$ where T_i are Chebychev polynomials and $x = F_c/F_{\max}$ (Prince, 1982; Watkin, 1994)
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.13$ e Å^{-3}
$\Delta\rho_{\min} = -1.04$ e Å^{-3}
Extinction correction: Larson (1970), Equation 22
Extinction coefficient: 8.2 (7)

Table 1
Selected geometric parameters (Å, °).

Br1—C16	1.949 (3)	N1—C9	1.417 (3)
Br2—C16	1.944 (3)	C2—C3	1.362 (3)
Br3—C17	1.950 (3)	C2—C16	1.479 (4)
Br4—C17	1.946 (3)	C3—C4	1.437 (4)
S1—O1	1.424 (2)	C3—C17	1.479 (4)
S1—O2	1.424 (2)	C4—C5	1.400 (4)
S1—N1	1.685 (2)	C4—C9	1.403 (4)
S1—C10	1.748 (3)	C8—C9	1.399 (4)
N1—C2	1.418 (3)		
O1—S1—O2	119.82 (13)	C3—C4—C5	132.6 (2)
O1—S1—N1	106.02 (12)	C3—C4—C9	107.1 (2)
O2—S1—N1	106.38 (12)	C5—C4—C9	120.2 (2)
O1—S1—C10	108.89 (14)	N1—C9—C4	107.8 (2)
O2—S1—C10	109.80 (13)	N1—C9—C8	130.6 (2)
N1—S1—C10	104.83 (12)	C4—C9—C8	121.5 (3)
S1—N1—C2	126.43 (18)	S1—C10—C11	119.5 (2)
S1—N1—C9	123.48 (18)	S1—C10—C15	118.8 (2)
C2—N1—C9	107.6 (2)	C11—C10—C15	121.6 (3)
N1—C2—C3	108.7 (2)	C2—C16—Br2	110.79 (18)
N1—C2—C16	122.8 (2)	C2—C16—Br1	111.52 (18)
C3—C2—C16	128.3 (2)	Br2—C16—Br1	110.79 (13)
C2—C3—C4	108.7 (2)	C3—C17—Br4	112.44 (18)
C2—C3—C17	123.9 (2)	C3—C17—Br3	110.96 (18)
C4—C3—C17	127.3 (2)	Br4—C17—Br3	110.18 (13)

The H atoms were included in the riding-model approximation, with C—H = 1.00 Å and $U_{\rm iso}(H) = 1.17–1.22U_{\rm eq}(C)$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT-NT* (Bruker, 2006); data reduction: *SAINT-NT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CRYSTALS*.

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