

## 3-Bromo-2-(4-bromo-2,5-dimethoxybenzyl)-1-phenylsulfonyl-1H-indole

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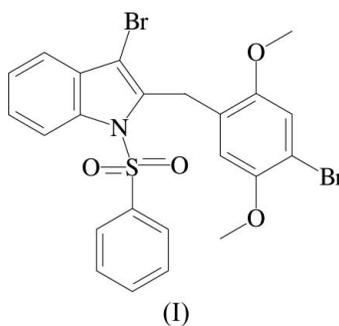
## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å  
 $R$  factor = 0.044  
 $wR$  factor = 0.116  
Data-to-parameter ratio = 19.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound,  $\text{C}_{23}\text{H}_{19}\text{Br}_2\text{NO}_4\text{S}$ , the orientation of the phenylsulfonyl substituent with respect to the indole ring system is influenced by intramolecular  $\text{C}-\text{H} \cdots \text{O}$  interactions. The sulfonyl-bound phenyl ring is orthogonal to the indole ring system. In the crystal structure,  $\pi-\pi$  stacking interactions involving the indole ring system link symmetry-related molecules into dimers.

## Comment

Polyhalogenated indole derivatives exhibit marked antimicrobial activity against Gram-positive and Gram-negative bacteria and fungi (Piscopo, Diurno, Mazzoni & Ciaccio, 1990; Piscopo, Diurno, Mazzoni, Ciaccio & Veneruso, 1990). 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). As part of our investigations on indole derivatives, we have undertaken the X-ray structure analysis of the title compound, (I).



The bond lengths and angles in (I) (Fig. 1) agree with those observed in other phenylsulfonylindoles (Ravishankar *et al.*, 2003*a,b*, 2005*a,b*; Malathy Sony *et al.*, 2005). As a result of the electron-withdrawing character of the phenylsulfonyl group, the  $\text{N}-\text{C}_{\text{sp}^2}$  bond lengths, *viz.*  $\text{N1}-\text{C1}$  [1.420 (4) Å] and  $\text{N1}-\text{C8}$  [1.425 (4) Å], are longer than the mean value of 1.355 (14) Å reported for N atoms with planar configurations (Allen *et al.*, 1987). Atom S1 has a distorted tetrahedral configuration, with angles  $\text{O1}-\text{S1}-\text{O2}$  [120.61 (16)°] and  $\text{N1}-\text{S1}-\text{C9}$  [105.02 (15)°] deviating significantly from ideal tetrahedral values.

The orientation of the phenylsulfonyl group with respect to the planar indole ring system (r.m.s. deviation = 0.011 Å) is influenced by intramolecular  $\text{C}-\text{H} \cdots \text{O}$  interactions (Table 1) involving the sulfonyl atoms O1 and O2; these atoms deviate by 0.163 (5) and 0.029 (5) Å, respectively, from the plane of the indole ring system. As shown in Fig. 1, each of these interactions generates rings of graph-set motif  $S(5)$  or  $S(6)$

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(Bernstein *et al.*, 1995; Etter, 1990). The dihedral angle between the indole ring system and the C9–C14 phenyl ring is  $89.2(1)^\circ$ . The 4-bromo-2,5-dimethoxybenzyl substituent is orthogonal to the indole ring system and twisted about the C15–C16 bond, as indicated by the torsion angles N1–C1–C15–C16 [ $89.5(4)^\circ$ ] and C1–C15–C16–C17 [ $23.4(5)^\circ$ ]. The C22–O3–C18–C17 [ $-8.8(6)^\circ$ ] and C23–O4–C21–C20 [ $-4.9(6)^\circ$ ] torsion angles indicate that the two methoxy substituents are essentially coplanar with the attached ring. The dihedral angle between the mean planes through the C9–C14 and C16–C21 aromatic rings is  $13.2(2)^\circ$ ; the centroids of these two rings are separated by  $4.063(2) \text{ \AA}$  and hence there is only a partial  $\pi$ - $\pi$  interaction between them [C9...C17 =  $3.559(5) \text{ \AA}$ , C10...C16 =  $3.426(5) \text{ \AA}$ , C10...C17 =  $3.396(5) \text{ \AA}$  and C11...C18 =  $3.542(6) \text{ \AA}$ ].

In the crystal structure, a  $\pi$ - $\pi$  stacking interaction involving the indole ring system links symmetry-related molecules at  $(x, y, z)$  and  $(1-x, y, \frac{1}{2}-z)$  into a dimer; the centroid-centroid distance between the indole ring systems is  $3.723(2) \text{ \AA}$  and the perpendicular distance is  $3.430(2) \text{ \AA}$ . A view of the molecular packing down the *b* axis, illustrating the dimer formation, is shown in Fig. 2. The dimers are linked by a weak intermolecular C–H...O interaction (Table 1). A Br1...O1( $x, 1+y, z$ ) short contact of  $3.248(3) \text{ \AA}$  is also observed.

## Experimental

To a solution of 3-bromo-1-phenylsulfonylindol-2-ylmethanol (25 mmol) in chloroform (400 ml), a solution of 4-bromo-3-methoxyacetanilide (25 mmol) in the same solvent (25 ml) was added, followed by anhydrous magnesium sulfate (10 g) and boron trifluoride etherate (2.0 ml). The resulting solution was refluxed for 3 h. Water (100 ml) was then added and the organic layer was separated. The organic layer was washed with 20% hydrochloric acid (50 ml), followed by water and saturated sodium bicarbonate solution. The solvent was removed by distillation, after drying over anhydrous sodium sulfate. The residue was chromatographed on a silica gel column (350 mesh) and eluted successively with 20, 25 and 30% ethyl acetate in hexane. The 30% ethyl acetate eluent gave the title compound, which was then crystallized from hexane–chloroform (2:1).

### Crystal data

$C_{23}H_{19}Br_2NO_4S$	$D_x = 1.659 \text{ Mg m}^{-3}$
$M_r = 565.27$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 3615 reflections
$a = 33.177(2) \text{ \AA}$	$\theta = 5.1\text{--}49.8^\circ$
$b = 9.0850(6) \text{ \AA}$	$\mu = 3.71 \text{ mm}^{-1}$
$c = 16.1712(10) \text{ \AA}$	$T = 293(2) \text{ K}$
$\beta = 111.802(1)^\circ$	Block, colourless
$V = 4525.5(5) \text{ \AA}^3$	$0.42 \times 0.24 \times 0.18 \text{ mm}$
$Z = 8$	

### Data collection

Siemens SMART CCD area-detector diffractometer	5536 independent reflections
$\omega$ scans	3263 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.041$
$T_{\text{min}} = 0.282$ , $T_{\text{max}} = 0.513$	$\theta_{\text{max}} = 28.3^\circ$
13791 measured reflections	$h = -41 \rightarrow 44$
	$k = -12 \rightarrow 11$
	$l = -21 \rightarrow 15$

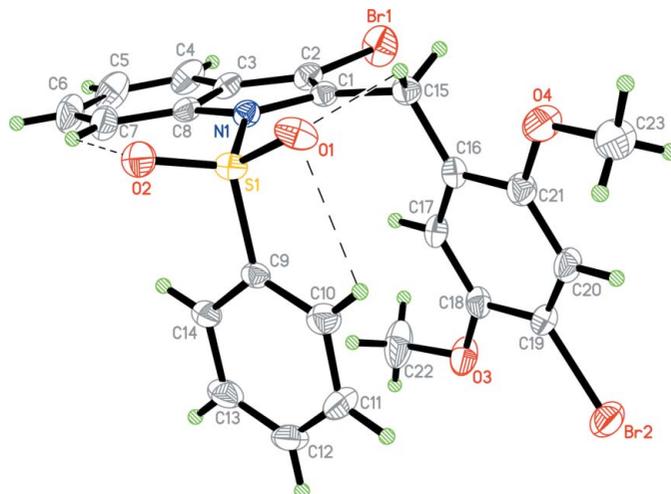


Figure 1

The structure of (I), showing the atom-numbering scheme and intra-molecular hydrogen bonds (dashed lines). Displacement ellipsoids are drawn at the 30% probability level.

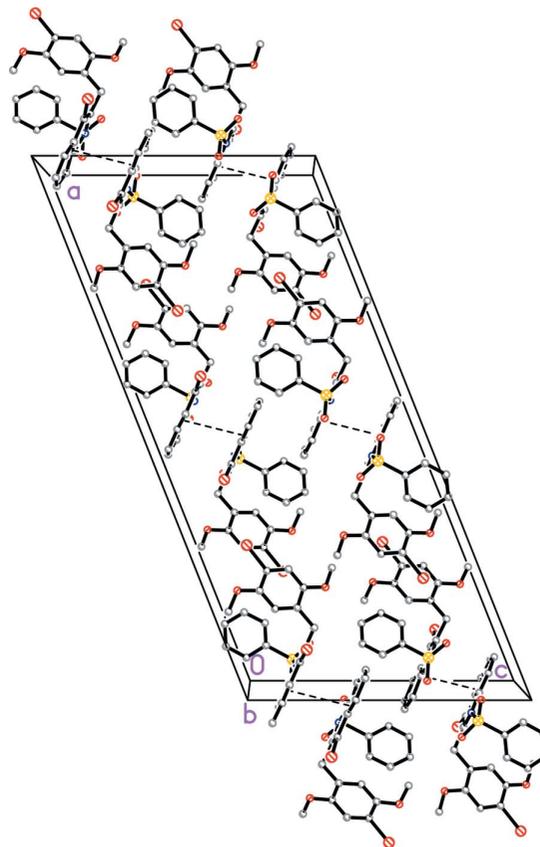


Figure 2

The crystal packing in (I), showing the dimers;  $\pi$ - $\pi$  interactions are shown as dashed lines.

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 5.2213P]$
$R[F^2 > 2\sigma(F^2)] = 0.044$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.116$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.89 \text{ e \AA}^{-3}$
5536 reflections	$\Delta\rho_{\text{min}} = -0.69 \text{ e \AA}^{-3}$
282 parameters	
H-atom parameters constrained	

**Table 1**  
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C7—H7 $\cdots$ O2	0.93	2.32	2.906 (5)	120
C10—H10 $\cdots$ O1	0.93	2.52	2.899 (5)	105
C15—H15A $\cdots$ O1	0.97	2.28	2.915 (5)	122
C12—H12 $\cdots$ O4 <sup>i</sup>	0.93	2.60	3.394 (6)	143

Symmetry code: (i)  $x, -y, z + \frac{1}{2}$

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINTE* (Siemens, 1996); data reduction: *SAINTE*; 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).

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