

2-Bromo-*N*-[3-bromo-*N*-(phenylsulfonyl)-indol-2-ylmethyl]aniline

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Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(\text{C}-\text{C}) = 0.018 \text{ \AA}$

R factor = 0.060

w*R* factor = 0.194

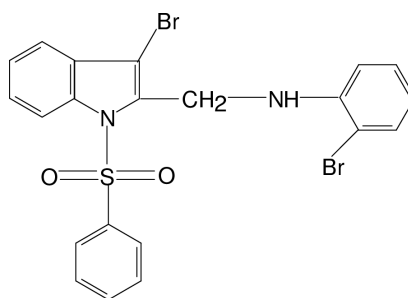
Data-to-parameter ratio = 13.2

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

In the title molecule, $\text{C}_{21}\text{H}_{16}\text{Br}_2\text{N}_2\text{O}_2\text{S}$, the S atom shows a distorted tetrahedral geometry. The sulfonylphenyl and bromophenyl rings are oriented almost perpendicular to the indole ring system, with dihedral angles of 84.2 (5) and 85.0 (5)°, respectively. The molecular structure is stabilized by C—H···O and N—H···Br intramolecular hydrogen bonds.

Comment

The indole ring system is present in a number of natural products, many of which are found to possess antibacterial (Okabe & Adachi, 1998), antitumour (Schollmeyer *et al.*, 1995), antidepressant (Papenstasion & Newmeyer, 1972), antimicrobial (El-Sayed *et al.*, 1986; Gadaginamath & Patil, 1999) and anti-inflammatory activities (Rodriguez *et al.*, 1985; Polleto *et al.*, 1974). Indole compounds exhibit physiological activities (Gilchrist, 1997; Marumo *et al.*, 1968; Shellard & Lala, 1978; Mukhopadhyay *et al.*, 1998; Takayama *et al.*, 1999). Indoles have also been proven to display high aldose reductase inhibitory activity (Rajeswaram *et al.*, 1999) and are found in numerous commercial products, such as pharmaceuticals, fragrances and dyes (Padwa *et al.*, 1999). Sulfonamides inhibit the growth of bacterial organisms and are also useful for treating urinary and gastrointestinal infections. In view of the above biological importance, we have undertaken the X-ray structure analysis of the title compound, (I).



(I)

In (I), the indole ring system is planar within ± 0.02 (1) Å. In the benzene ring of the indole moiety, the endocyclic C—C angles at atoms C4 and C7 are narrowed to 118.5 (13) and 114.8 (12)°, while those at C5 and C8 are widened to 122.0 (14) and 123.0 (11)°, respectively. This effect may be caused by the fusion of the smaller pyrrole ring to the six-membered benzene ring (Allen, 1981). A similar effect has also been observed by both Varghese *et al.* (1986) and Sethusankar *et al.* (2002). The S atom is in a distorted tetrahedral configuration. The widening of the O1—S—O2 angle to 118.6 (5)°, and the resultant narrowing of the C14—S—N1 angle to 105.6 (5)°, from the ideal tetrahedral value, are

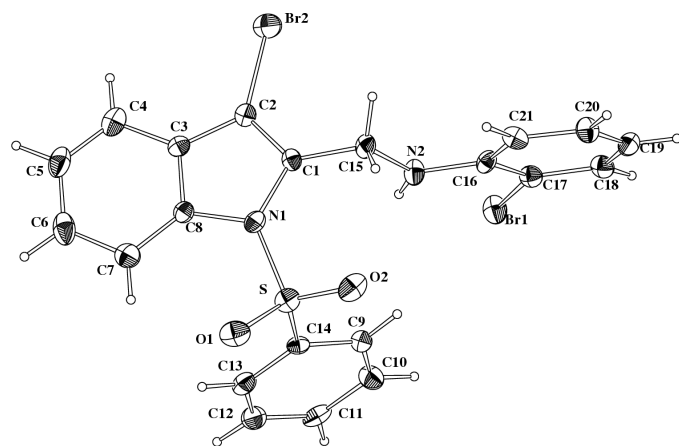


Figure 1
The molecular structure of (I). Displacement ellipsoids are shown at the 50% probability level.

attributed to the Thorpe–Ingold effect (Bassindale, 1984; Sethusankar *et al.*, 2002). The O1–S–N1–C1 [156.3 (9)°] and O1–S–C14–C13 [25.0 (10)°] torsion angles describe the conformation of the phenylsulfonyl group with respect to the indole moiety; the mean planes through the indole and phenyl rings form a dihedral angle of 84.2 (4)°, as observed in similar structures (Yokum & Fronczek, 1997; Sankaranarayanan *et al.*, 2000). The phenyl ring of the *o*-bromoaniline group forms a dihedral angle of 18.7 (5)° with the phenyl ring of the phenylsulfonyl group. The N1–S–C14–C9 and N1–S–C14–C13 torsion angles are 92.4 (9) and –88.5 (9)°, respectively, and lie within the range 70–120° (Gomes *et al.*, 1993). The relatively large values of the C–N distances in the indole moiety, namely N1–C1 and N1–C8 [1.424 (13) and 1.444 (13) Å, respectively], are due to the electron-withdrawing character of the phenylsulfonyl group (Govindasamy *et al.*, 1997, 1998). The S–N [1.679 (9) Å] and S–C [1.764 (11) Å] distances agree with previously reported values (Allen *et al.*, 1987; Yokum & Fronczek, 1997). The molecular structure is stabilized by C–H···O and N–H···Br intramolecular hydrogen bonds (Table 2).

Experimental

A solution of 1-phenylsulfonyl-2-bromomethyl-2-bromoindole (4.29 g, 10 mmol) and *o*-bromoaniline (2 equivalents) in dry DMF (25 ml) containing finely powdered K₂CO₃ (200 mg) was stirred at room temperature for 12 h. The reaction mixture was poured on to ice (200 g) and the solid which formed was filtered off immediately and washed with an excess of water. The crude product was dried over CaCl₂ and recrystallized from ethyl acetate–hexane (1:9) to give colourless crystals of the title compound.

Crystal data

C₂₁H₁₆Br₂N₂O₂S
M_r = 520.24
Monoclinic, *P*₂₁/*c*
a = 12.3288 (9) Å
b = 8.4100 (8) Å
c = 19.441 (2) Å
β = 106.296 (7)°
V = 1934.7 (3) Å³
Z = 4

D_x = 1.786 Mg m^{−3}
Mo *K*α radiation
Cell parameters from 25 reflections
θ = 1.7–25.0°
μ = 4.32 mm^{−1}
T = 293 (2) K
Prism, colourless
0.40 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Non-profiled ω/2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
*T*_{min} = 0.277, *T*_{max} = 0.357
3502 measured reflections
3389 independent reflections
1306 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.095
θ_{max} = 25.0°
h = −14 → 14
k = 0 → 9
l = 0 → 23
3 standard reflections
frequency: 300 min
intensity decay: 1%

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.060
wR [*F*²] = 0.194
S = 0.87
3389 reflections
257 parameters

H atoms treated by a mixture of independent and constrained refinement
w = 1/[σ²(*F*_o²) + (0.1*P*)²]
where *P* = (*F*_o² + 2*F*_c²)/3
(Δ/σ)_{max} < 0.001
Δρ_{max} = 0.59 e Å^{−3}
Δρ_{min} = −0.62 e Å^{−3}

Table 1

Selected geometric parameters (Å, °).

S–N1	1.679 (9)	N1–C1	1.424 (13)
S–C14	1.764 (11)	N1–C8	1.441 (13)
O2–S–O1	118.6 (5)	C4–C5–C6	122.0 (14)
N1–S–C14	105.6 (5)	C7–C8–C3	123.0 (11)
N1–C1–C15	123.7 (10)	N2–C15–C1	112.4 (9)
C5–C4–C3	118.5 (13)		
O1–S–N1–C1	156.3 (9)	N1–S–C14–C9	92.4 (9)
O1–S–C14–C13	25.0 (10)	N1–C1–C15–N2	88.3 (12)
N1–S–C14–C13	−88.5 (9)		

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
N2–H2···Br1	0.90 (9)	2.66 (9)	3.054 (9)	108 (7)
C15–H15B···O2	0.97	2.33	2.888 (14)	116
C7–H7···O1	0.93	2.36	2.938 (16)	120

The H atom attached to N2 was located from a difference map and both positional and isotropic displacement parameters were refined [*N*–H = 0.90 (9) Å]. All other H atoms were fixed geometrically and allowed to ride on the parent C atoms. The reflection (006) was removed during refinement, as the observed and calculated structure factors showed large disagreement. Owing to the poor diffraction quality of the crystal, the ratio of observed to unique reflections is low (39%) and the *R*_{int} value is high (0.095).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1983, 1995).

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