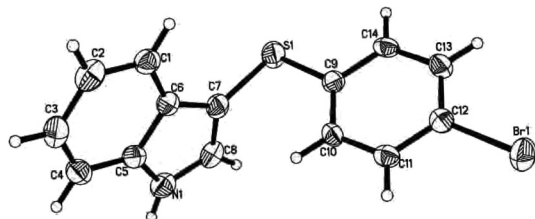


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Crystal structure of 3-((4-bromophenyl)thio)-1H-indole, $C_{14}H_{10}BrNS$



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Abstract

$C_{14}H_{10}BrNS$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 5.6392(2)$ Å, $b = 7.7930(3)$ Å, $c = 28.2476(8)$ Å, $V = 1241.37(8)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0414$, $wR_{ref}(F^2) = 0.1079$, $T = 293$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

To a solution of indole (0.1171 g, 1 mmol) and 4-bromothiophenol (0.1891 g, 1 mmol) in DCE (5 mL) and DMSO (1 mL) was added I_2 (0.8438 g, 5 mmol %). The mixture was stirred at 60 °C under oxygen atmosphere for 6 h. The reaction mixture was filtered and the residue was extracted with ethyl acetate (15 mL) for three times. The combined organic layers were washed with water and brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. After removal of

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	$0.15 \times 0.12 \times 0.10$ mm
Wavelength:	Cu $K\alpha$ radiation (1.54184 Å)
μ :	5.87 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
θ_{max} , completeness:	70.3° , >99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4577, 2320, 0.033
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2077
$N(\text{param})_{\text{refined}}$:	159
Programs:	CrysAlis ^{pro} [1], Olex2 [2], SHELX [3]

the solvent, the residue was then purified by chromatography column to give the desired (271 mg, 89 %) as a white solid. Single crystals were obtained by crystallization from ethyl acetate/petroleum. **Melting point:** 140–142 °C. $^1\text{H NMR}$ (400 MHz, $CDCl_3$, 298 K) δ 8.46 (s, 1H), 7.57 (d, $J = 8.2$ Hz, 1H), 7.50 (d, $J = 2.5$ Hz, 1H), 7.45 (d, $J = 8.2$ Hz, 1H), 7.31–7.24 (m, 3H), 7.21–7.15 (m, 1H), 6.99–6.92 (m, 2H). $^{13}\text{C NMR}$ (101 MHz, $CDCl_3$, 298 K) δ 138.7, 136.7, 131.8, 130.9, 128.9, 127.5, 123.4, 121.2, 119.7, 118.4, 111.8, 102.5.

2 Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

Indoles and their derivatives are ubiquitously found in a diverse array of biologically active molecules. One of them is 3-sulfinylindole, which exhibits excellent pharmacological properties, including anti-HIV [4, 5], antinociceptive [6], and anti-allergic activities [7], and acting as 5-lipoxygenase and tubulin polymerization inhibitor [8, 9]. The essence of 3-sulfinylindole synthesis is the synthesis of compounds containing C–S bonds by oxidative coupling reactions of indoles with thiol derivatives under suitable conditions. The construction of C–S bonds represents an important step in organic synthesis [10, 11]. In recent years, direct sulfonylation through C–H functionalization has attracted remarkable attention and has become an efficient strategy for

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Br1	0.45857 (16)	0.36761 (10)	0.84451 (2)	0.0673 (3)
S1	0.0288 (4)	0.4675 (3)	0.63551 (6)	0.0661 (5)
N1	0.5458 (11)	0.3366 (8)	0.54910 (19)	0.0602 (14)
H1	0.676 (8)	0.358 (9)	0.534 (2)	0.06 (2)*
C1	0.0700 (12)	0.0624 (9)	0.5836 (2)	0.0524 (15)
H1A	−0.0652	0.0715	0.6023	0.063*
C2	0.1247 (14)	−0.0899 (9)	0.5607 (2)	0.0619 (19)
H2	0.0264	−0.1847	0.5645	0.074*
C3	0.3243 (14)	−0.1032 (9)	0.5318 (2)	0.0614 (18)
H3	0.3557	−0.2068	0.5168	0.074*
C4	0.4756 (13)	0.0324 (8)	0.5251 (2)	0.0558 (16)
H4	0.6072	0.0228	0.5054	0.067*
C5	0.4255 (11)	0.1841 (8)	0.54867 (19)	0.0464 (14)
C6	0.2227 (11)	0.2013 (8)	0.57807 (19)	0.0445 (13)
C7	0.2325 (11)	0.3721 (9)	0.5966 (2)	0.0511 (14)
C8	0.4272 (14)	0.4494 (9)	0.5784 (2)	0.0581 (17)
H8	0.4741	0.5615	0.5848	0.070*
C9	0.1549 (11)	0.4294 (7)	0.6921 (2)	0.0442 (13)
C10	0.3644 (11)	0.3431 (8)	0.6999 (2)	0.0483 (14)
H10	0.4483	0.2984	0.6744	0.058*
C11	0.4515 (12)	0.3222 (7)	0.7455 (2)	0.0475 (13)
H11	0.5928	0.2637	0.7507	0.057*
C12	0.3253 (11)	0.3897 (7)	0.7828 (2)	0.0451 (13)
C13	0.1155 (11)	0.4733 (8)	0.7763 (2)	0.0507 (15)
H13	0.0324	0.5169	0.8021	0.061*
C14	0.0277 (11)	0.4921 (7)	0.7307 (2)	0.0472 (13)
H14	−0.1167	0.5470	0.7259	0.057*

constructing complicated organosulfur compounds. Meanwhile, this contribution is part of our continuing interest in synthesis and understanding of schemes for the formation of C–S bonds between indoles and thiols.

The asymmetric unit contains one molecule of the title compound, which is constructed by the indole and the 4-bromothiophenol molecules (see the figure). The indole together with sulfur atom is almost in a strict plane, whereby the largest deviation for the S1 atom from the indole plane is 0.063 Å. The dihedral angle between the indole group and 4-bromothiophenol were found to be 86.53°. The C(9)–S(1)–C(7)–C(6) and C(9)–S(1)–C(7)–C(8) torsion angles are 93.7(5)° and −86.8(2)°. The C(7)–S(1)–C(9) bond angle is 103.4(3)°. The thioether bond distances are 1.756(7) Å for C(7)–S(1) and 1.775(6) Å for C(9)–S(1), respectively. Within the indole unit, the dimensions and planarity are consistent with their adoption of a localized enol form. The bond lengths of C(7)–C(6), C(7)–C(8) are 1.431(9) and 1.353(6) Å respectively. The structure of the molecule is similar to the stereo-configuration of the compound reported in the references. The bond lengths and angles are all in the expected ranges [12, 13]. And no unusual intermolecular contacts, were observed in this crystal.

The complete set of X-ray diffraction data for the title compound was deposited to the Cambridge Crystallographic Data Centre (CCDC entry no. 2294356).

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