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2,3-Diphenyl-1-(phenylsulfonyl)indole

Yanbing Liu,[a] Gordon W. Gribble[a] and Jerry P. Jasinski[b]*

[a]Department of Chemistry, Dartmouth College, Hanover, NH 03755-3564, USA, and
[b]Department of Chemistry, Keene State College, Keene, NH 03435-2001, USA

Correspondence e-mail: jjasinski@keene.edu

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The crystal structure of the title compound, $C_{26}H_{19}NO_2S$, reveals that the phenylsulfonyl ring and the adjacent phenyl ring are approximately *syn*–parallel to each other, with a dihedral angle of 18.4 (4)°.

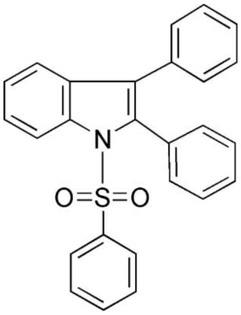
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.016 Å
R factor = 0.051
wR factor = 0.264
Data-to-parameter ratio = 13.7

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

Comment

Diarylindoles and other diarylheterocycles have significant anti-inflammatory activity (Szmuszkovicz *et al.*, 1966). For example, 2,3-bis(4-methoxyphenyl)indole (Indoxole), the discovery of which paved the way for the development of Celebrex and Vioxx (McAdam *et al.*, 1999), has powerful anti-arthritic and antipyretic activity, comparable in some assays with aspirin and indomethacin (Klug *et al.*, 1981). In 2000, we reported a synthesis of 2,3-diarylindoles that employs a bis-Suzuki reaction of 2,3-dihaloindoles (Liu & Gribble, 2000), and we now describe the X-ray crystal structure determination of the title compound (I).



(I)

The observed geometric parameters are generally comparable with the reported values for other *N*-(phenylsulfonyl)indoles (Beddoes *et al.*, 1986; Schollmeyer *et al.*, 1995; Yokum & Fronczek, 1997; Govindasamy *et al.*, 1998; Simon *et al.*, 2000; Sankaranarayanan *et al.*, 2000; Sonar *et al.*, 2004; Palani *et al.*, 2006). The indole ring system is essentially planar, with a mean deviation for the nine ring atoms of 0.011 (8) Å. The S—N, S—C and S═O distances of 1.682 (8), 1.746 (10) and 1.424 (7) Å, respectively, are comparable with those observed in other *N*-(phenylsulfonyl)indoles and in *N*-phenylsulfonamides (Gomes *et al.*, 1993; Palani *et al.*, 2006).

Most interesting is that the phenylsulfonyl benzene ring and the phenyl ring attached to C2 are approximately *syn*–parallel to each other, with a dihedral angle of 18.4 (4)°, which is perhaps indicative of some π-interaction. Consequently, the sulfonyl group does not adopt the usual nitrogen-sulfonyl geometry seen with other *N*-(phenylsulfonyl)indoles. As a result, the normally observed geometry of the *N*-phenyl-

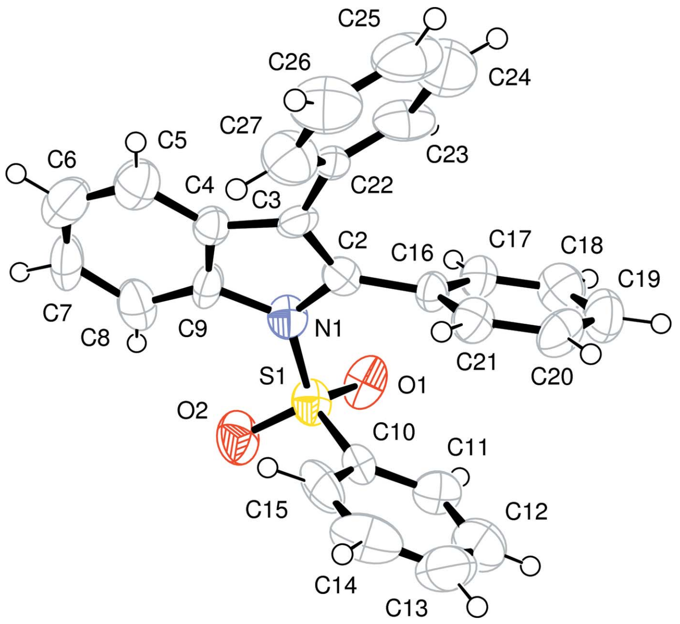


Figure 1

The molecular structure of (I), showing the atom-labeling scheme and with 50% probability displacement ellipsoids.

sulfonyl group, wherein the N lone pair eclipses the sulfonyl group in *N*-(phenylsulfonyl)indoles (Beddoes *et al.*, 1986), is not observed with (I). Accordingly, whereas in (I) the O2—S1—N1—C9 torsion angle is only −1.0 (8)°, the O1—S1—N1—C2 torsion angle is −75.4 (7)°. For comparison with the latter angle, the corresponding O1—S1—N1—C2 torsion angle in *N*-(phenylsulfonyl)indole is −37° and that in *N*-(phenylsulfonyl)pyrrole is −35° (Beddoes *et al.*,1986). Furthermore, the dihedral angle in (I) between the indole ring system and the benzene ring of the phenylsulfonyl group is only 66.2 (4)°, instead of the more typical 80–95° [e.g. 86.80 (6)° (Palani *et al.*, 2006), 82.83 (5)° (Yokum & Fronczek, 1997), 85.6 (1)° (Sankaranarayanan *et al.*, 2000), and 90.2 (2)° and 94.0 (2)° (Sonar *et al.*, 2004)], which presumably reflects interaction with the phenyl ring attached to C2. The phenyl rings attached to C2 and C3 are also twisted out of the indole ring plane at angles of 60.5 (3) and 132.9 (4)°, respectively. For comparison, the corresponding phenyl torsion angles in 2,3-diphenylindole are 44.0 and 54.1°, respectively (Schmelter *et al.*, 1973).

Experimental

A mixture of 2,3-diiodo-1-(phenylsulfonyl)indole (0.100 g, 0.196 mmol), phenylboronic acid (0.432 mmol), palladium(II) acetate (4.4 mg, 0.0196 mmol), tri(*o*-tolyl)phosphine (12 mg, 0.039 mmol) and potassium carbonate (0.33 g, 2.4 mmol) in a three-necked round-bottomed flask was flushed with nitrogen for 10 min. A solution of acetone (10 ml) and water (5 ml) was added by syringe and the mixture was heated at 343K (reflux) for 5 h under nitrogen (monitored by thin-layer chromatography). The cooled reaction mixture was extracted with diethyl ether. The ether layer was dried ($MgSO_4$) and evaporated to afford the crude product. Flash chromatography over silica gel (EtOAc–hexane, 30:70 *v*/*v*) gave (I) in 83% yield. Recrystallization from mixtures of diethyl ether and hexane (1:1) afforded an analytical sample (m.p. 447–449 K). HRMS, *m*/*z* calculated for $C_{26}H_{19}NO_2S$ (M^+) 409.1137, found 409.1138. Analysis, calculated for $C_{26}H_{19}NO_2S$: C 76.26, H 4.68, N 3.42, S 7.83%; found: C 76.34, H 4.73, N 3.50, S 7.82%.

Crystal data

$C_{26}H_{19}NO_2S$
$M_r = 409.48$
Monoclinic, $P2_1/c$
$a = 11.768$ (6) Å
$b = 15.198$ (4) Å
$c = 12.223$ (7) Å
$\beta = 111.71$ (3)°
$V = 2030.9$ (16) Å^3
$Z = 4$
$D_x = 1.339$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.18$ mm^{-1}
$T = 296$ (2) K
Plate, colorless
0.40 × 0.40 × 0.10 mm

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.931$, $T_{\max} = 0.982$
3716 measured reflections
3716 independent reflections
789 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 27.5$°
3 standard reflections every 150 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.264$
$S = 0.92$
3716 reflections
272 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0942P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}

The H atoms were included in the riding-model approximation, with C—H = 0.93 Å, and with $U_{\rm iso}({\rm H}) = 1.11–1.20U_{\rm eq}({\rm C})$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *CrystalStructure* (Rigaku, 2005); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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