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1-(Phenylsulfonyl)-2,3-di-*p*-tolyl-1*H*-indole

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The crystal structure of the title compound, $C_{28}H_{23}NO_2S$, reveals that the phenylsulfonyl ring and the adjacent benzene ring are *anti*, with no evidence of π stacking.

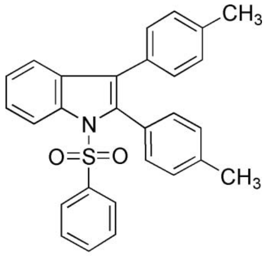
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.006 Å
R factor = 0.051
wR factor = 0.211
Data-to-parameter ratio = 18.1

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

Comment

Liu *et al.* (2007) described the crystal structure of 2,3-diphenyl-1-(phenylsulfonyl)indole derived from a bis-Suzuki reaction on 2,3-diiodo-1-(phenylsulfonyl)indole (Liu & Gribble, 2000). In continuation of our interest in these anti-inflammatory compounds (Szmuszkovicz *et al.*, 1966; Klug *et al.*, 1981), we now describe the X-ray structure determination of the 2,3-di-*p*-tolyl derivative (I), which was similarly synthesized *via* a double Suzuki coupling reaction. The observed parameters are generally 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; Simon *et al.*, 2000; Sankaranarayanan *et al.*, 2000; Sonar *et al.*, 2004; Palani *et al.*, 2006; Liu *et al.*, 2007).



(I)

The indole ring system is essentially planar with a mean deviation for the nine ring atoms of 0.018 (3) Å. The sum of the bond angles surrounding the N atom is only 253.1°, indicative of pyramidalization, in contrast to the normal sp^2-hybridization observed for 1-(phenylsulfonyl)indole itself (Beddoes *et al.*, 1986). The S—N, S—C, and S═O distances of 1.675 (2), 1.759 (4), and 1.416 (4) Å, respectively, are comparable to those observed in other *N*-(phenylsulfonyl)indoles and in *N*-phenylsulfonamides (Beddoes *et al.*, 1986; Gomes *et al.*, 1993; Palani *et al.*, 2006; Liu *et al.*, 2007). The C2—C3 indole double bond length in (I) of 1.355 (4) Å is longer than that in *N*-(phenylsulfonyl)indole itself [1.336 (3) Å; Beddoes *et al.*, 1986], presumably as a result of steric repulsion between the C2 and C3 benzene rings.

The benzene ring of the phenylsulfonyl group makes a dihedral angle with the mean plane of the indole ring of 101.0 (2)°, instead of the more typical 80–95°, which presumably reflects steric interaction with the C2 benzene group. The C2 and C3 benzene rings are twisted away from the indole ring

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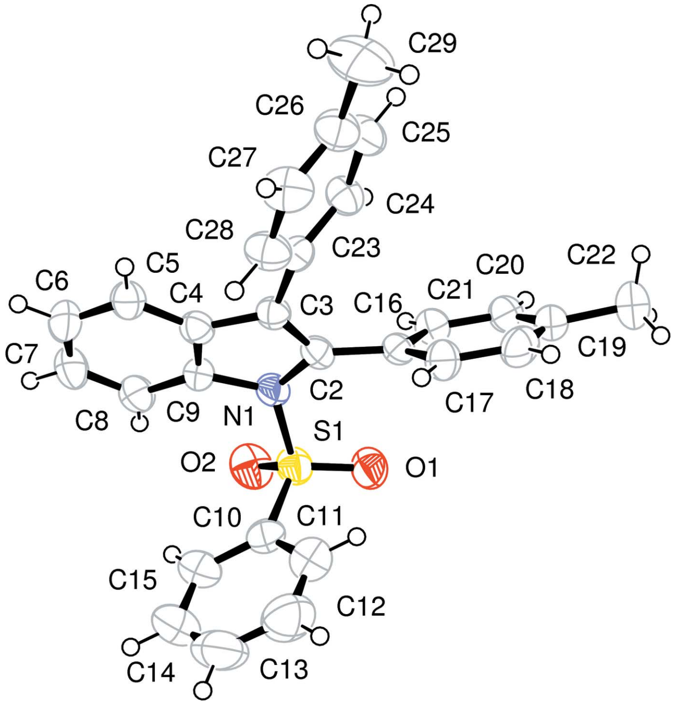

Figure 1
Molecular structure of (I), showing the atom labeling and 50% probability displacement ellipsoids.

plane with torsion angles of 126.0 (1) and 124.7 (1)°, respectively. For comparison, the corresponding C2 and C3 phenyl torsion angles in 2,3-diphenylindole are 44.0 and 54.1°, respectively (Schmelter, *et al.*, 1973). The interaction between the C2 benzene group and the phenylsulfonyl group in (I) forces the sulfonyl group not to adopt the usual nitrogen–sulfonyl geometry seen with other *N*-(phenylsulfonyl)indoles wherein the N lone pair eclipses the sulfonyl group in *N*-(phenylsulfonyl)indoles (Beddoes *et al.*, 1986). Accordingly, the O2—S1—N1—C9 torsion angle is 53.4 (3)° and the O1—S1—N1—C2 torsion angle is −29.2 (3)°. However, there is more distortion in 2,3-diphenyl-1-(phenylsulfonyl)indole for which the O2—S1—N1—C9 torsion angle is −1.0 (8)° and the O1—S1—N1—C2 torsion angle is −75.4 (7)° (Liu *et al.*, 2007). For comparison, the corresponding O1—S1—N1—C2 torsion angle in *N*-(phenylsulfonyl)indole (Beddoes *et al.*, 1986) is −37° and in *N*-(phenylsulfonyl)pyrrole it is −35° (Beddoes *et al.*, 1986). Interestingly, whereas 2,3-diphenyl-1-(phenylsulfonyl)indole adopts a *syn* geometry for the C2 phenyl and phenylsulfonyl rings (Liu *et al.*, 2007), an *anti* geometry is observed with (I).

Experimental

A mixture of 2,3-diiodo-1-(phenylsulfonyl)indole (0.100 g, 0.196 mmol), *p*-tolylboronic 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 343 K (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) gave (I) in 98% yield. Recrystallization from mixtures of diethyl ether and hexane (1:1) afforded an analytical sample; m.p. 426–428 K; HRMS *m*/*z* calculated for $C_{28}H_{23}NO_2S$ (M^+): 437.1450; found: 437.1446. Analysis calculated for $C_{28}H_{23}NO_2S$: C 76.86, H 5.30, N 3.20, S 7.33%; found: C 76.98, H 5.37, N 3.26, S 7.30%.

Crystal data

$C_{28}H_{23}NO_2S$
$M_r = 437.53$
Triclinic, $P\overline{1}$
$a = 9.521\ (2)$ Å
$b = 10.775\ (4)$ Å
$c = 12.060\ (4)$ Å
$\alpha = 74.61\ (3)^\circ$
$\beta = 102.14\ (3)^\circ$
$\gamma = 103.45\ (2)^\circ$
$V = 1145.6\ (6)$ Å^3
$Z = 2$
$D_x = 1.268$ Mg m^{-3}
Mo *Kα* radiation
$\mu = 0.17$ mm^{-1}
$T = 296$ K
Prism, colorless
0.50 × 0.40 × 0.20 mm

Data collection

Rigaku AFC-6S diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan
(North *et al.*, 1968)
$T_{\min} = 0.921$, $T_{\max} = 0.968$
5268 measured reflections
5268 independent reflections
1835 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 27.5^\circ$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.211$
$S = 0.97$
5268 reflections
291 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0946P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.009 (3)

The H atoms were included in the riding model approximation with C—H = 0.93–0.96 Å, and with $U_{\rm iso}(H) = 1.19–1.20U_{\rm eq}(C)$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *CrystalStructure* (Rigaku/MSC, 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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