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2-Nitro-1-(phenylsulfonyl)-1*H*-indole

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Key indicators

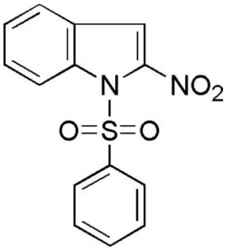
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.058
wR factor = 0.124
Data-to-parameter ratio = 18.9

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

The crystal structure of the title molecule, $C_{14}H_{10}N_2O_4S$, confirms the position of the nitro group at C-2 of the indole ring. The nitro group is not coplanar with the indole ring system and the angle between the planes of the indole ring system and the phenyl ring is 68.6 (1)°.

Comment

Roy, Pelkey *et al.* (2007) described the crystal structure determination of 3-nitro-1-(phenylsulfonyl)-1*H*-indole. The related 2-nitroindoles are important building blocks for the synthesis of pyrrolo[3,4-*b*]indoles *via* 1,3-dipolar cycloaddition reactions with azomethine ylides (Roy, Kishbaugh *et al.*, 2007) and mesoionic münchnones (1,3-oxazolium-5-olates) (Gribble *et al.*, 1998, 2000). Furthermore, the chemistry of these electron-deficient 2-nitroindoles is of great interest and utility for the synthesis of carbazoles *via* Diels–Alder cycloaddition reactions (Kishbaugh & Gribble, 2001) and for reduction to aminoindoles (Roy & Gribble, 2006). In connection with our interest in the crystal structures of nitroindoles (Roy, Pelkey *et al.*, 2007; Kishbaugh *et al.*, 2006), we now describe the crystal structure determination of 2-nitro-1-(phenylsulfonyl)-1*H*-indole, (I).



(I)

X-ray crystallographic analysis confirms the molecular structure and atom connectivity for (I) (Fig. 1). The indole ring system is planar, with bond distances and angles in agreement with other indole derivatives (Beddoes *et al.*, 1986; Zarza *et al.*, 1988; Yokum & Fronczek, 1997; Seshadri *et al.*, 2002; Mason *et al.*, 2003; Gallagher *et al.*, 2004; Sonar *et al.*, 2004; Kishbaugh *et al.*, 2006; Roy, Pelkey *et al.*, 2007).

Unlike 3-nitro-1-(phenylsulfonyl)indole (Roy, Pelkey *et al.*, 2007), the nitro group of (I) is not coplanar with the indole ring system, and the sulfonyl is twisted away from the nitro group, indicating repulsion between the two. The values of the O4—N2—C2—N1 and O3—N2—C2—C3 torsion angles of 160.8 (2) and 149.8 (2)°, respectively, reveal the twisting of the nitro group out of the indole plane. The angle between the planes of the indole ring system and the phenyl ring is

68.6 (1)°. By comparison, this angle in 3-nitro-1-(phenylsulfonyl)-1*H*-indole is 83.9 (3)° (Roy, Pelkey *et al.*, 2007) and it is 91.72° in 1-(phenylsulfonyl)indole (Beddoes *et al.*, 1986).

The sum of the angles around indole atom N1 is 353.9°, indicating some deviation from sp^2 hybridization. The sum of the bond angles surrounding the nitro group atom N2 is 360.0°, indicating sp^2 hybridization.

The N2—C2 bond [1.431 (2) Å] is longer than this bond in 3-nitro-1-(phenylsulfonyl)-1*H*-indole (Roy, Pelkey *et al.*, 2007), indicating less resonance interaction between the indole ring system and the nitro group than is seen with 3-nitro-1-(phenylsulfonyl)-1*H*-indole, as expected from the known chemistry of 1-(phenylsulfonyl)indole, which favours reactivity at C3 (Pelkey & Gribble, 1999).

Experimental

To a stirred suspension of sodium hydride (60% dispersion in mineral oil, 94 mg, 2.34 mmol) in anhydrous dimethylformamide (5 ml) at 273 K was added a solution of 2-nitroindole (250 mg, 1.56 mmol) (Roy & Gribble, 2006) dissolved in anhydrous dimethylformamide (3 ml). The reaction mixture was stirred at 273 K for 30 min and then benzenesulfonyl chloride (0.23 ml, 1.75 mmol) was added dropwise. The reaction mixture was stirred at 273 K for 3 h and then at room temperature for 12 h. It was then poured on to ice and extracted with ethyl acetate (3 × 20 ml). The combined organic extracts were washed with brine, dried (Na_2SO_4) and concentrated to yield a red oil, which was purified by column chromatography (hexanes–dichloromethane, 2:1 *v*/*v*) to afford (I) as a yellow solid [yield 255 mg, 54%; m.p. 431–432 K (literature m.p. 430–433 K; Pelkey & Gribble, 1997)]. Recrystallization from hexanes–dichloromethane (2:1 *v*/*v*) afforded crystals of (I) suitable for X-ray analysis.

Crystal data

$C_{14}H_{10}N_2O_4S$
$M_r = 302.30$
Orthorhombic, *Pbca*
$a = 16.7613 (16)$ Å
$b = 7.4192 (5)$ Å
$c = 20.856 (2)$ Å
$V = 2593.6 (4)$ Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 0.27$ mm^{-1}
$T = 100 (2)$ K
0.42 × 0.28 × 0.27 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: numerical (*ABSCOR*; Higashi, 1999)
$T_{min} = 0.896$, $T_{max} = 0.931$
17003 measured reflections
4394 independent reflections
3766 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.124$
$S = 1.16$
4394 reflections
232 parameters
All H-atom parameters refined
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; 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*.

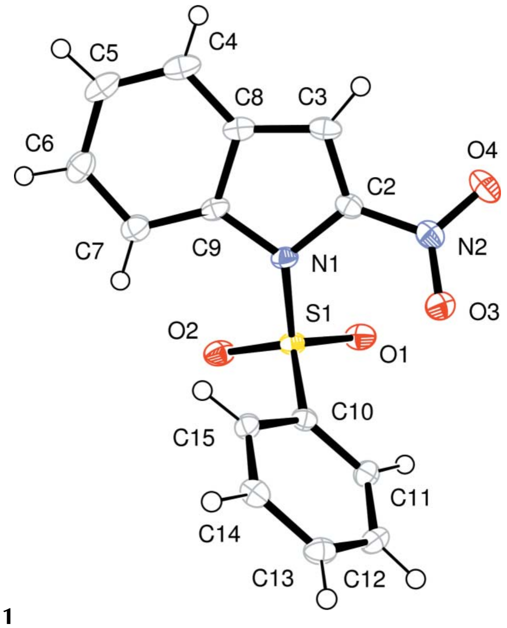


Figure 1
The molecular structure of (I), showing the atom labelling and with 50% probability displacement ellipsoids.

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