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9-(Phenylsulfonyl)-9*H*-carbazol-2-ol

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.008 Å
R factor = 0.056
wR factor = 0.191
Data-to-parameter ratio = 16.0

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

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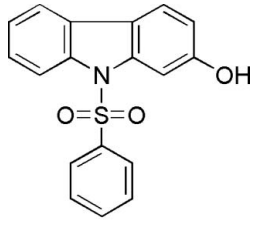
The crystal structure of the title molecule, $C_{19}H_{13}NO_3S$, confirms the structure of this compound formed from a Diels–Alder reaction between 3-nitro-1-(phenylsulfonyl)indole and 1-methoxy-3-(trimethylsiloxy)-1,3-butadiene (Danishefsky's diene). The angle between planes of tthe carbazole ring system and the phenylsulfonyl ring is 89.97 (17)°.

Comment

We have found that both 2- and 3-nitroindoles undergo Diels–Alder cycloaddition reactions to afford carbazoles (Kishbaugh & Gribble, 2001). As described in the preceding paper (Kishbaugh *et al.*, 2007), the reaction between 3-nitro-1-(phenylsulfonyl)indole and 1-methoxy-3-(trimethylsiloxy)-1,3-butadiene (Danishefsky's diene) provides the title compound, 9-(phenylsulfonyl)-9*H*-carbazol-2-ol, (I), as the minor product. Due to the anticipated spectroscopic similarity of 9-(phenylsulfonyl)-9*H*-carbazol-2-ol and the isomeric 9-(phenylsulfonyl)-9*H*-carbazol-3-ol, which could also be a Diels–Alder product of the described reaction, we deemed it necessary to determine the crystal structure of (I), which we now report.



(I)

The X-ray crystallographic analysis confirms the molecular structure and atom connectivity for (I) (Fig. 1) that we had proposed for this compound based on the structure of the initial cycloadduct (Kishbaugh & Gribble, 2001; Kishbaugh *et al.*, 2007). The carbazole ring is essentially planar and the angle between the planes of the carbazole ring system and the phenylsulfonyl ring is 89.97 (17)°, compared with an angle of 85.1 (7)° reported for 9-(*p*-tolylsulfonyl)-9*H*-carbazole (Li *et al.*, 2006). The hydroxyl group is in the plane of the carbazole and the sum of the bond angles surrounding the N atom is 356.1°, indicating slight pyramidalization, as was also the case for 9-(*p*-tolylsulfonyl)-9*H*-carbazole (357.88°; Li *et al.*, 2006).

Experimental

A solution of 3-nitro-1-(phenylsulfonyl)indole (187 mg, 0.62 mmol) (Pelkey & Gribble, 1999) and commercially available *trans*-1-meth-

oxy-3-(trimethylsilyloxy)-1,3-butadiene (0.2 ml, 0.93 mmol) in toluene (12 ml) was heated to reflux for 36 h and allowed to cool to room temperature. The toluene was removed by rotary evaporation. The resulting amorphous solid was dissolved in tetrahydrofuran (10 ml) and 10% aqueous HCl (5 ml) and stirred for 3 h. After removal of tetrahydrofuran by rotary evaporation, the solution was neutralized by the addition of 5% aqueous $NaHCO_3$ and extracted with dichloromethane (4 × 20 ml). The combined organic layers were washed with brine (20 ml), dried (Na_2SO_4) and concentrated, to yield a brown oil. This was purified by column chromatography (silica gel; hexanes–ethyl acetate 2:1 *v*/*v*) to yield (I) as a yellow solid (56 mg, 28%; m.p. 468–469 K). IR (film): λ_{max} 3347 (OH), 1367 (SO_2), 1176 (SO_2) cm^{-1}. Recrystallization from diethyl ether yielded yellow prisms of (I).

Crystal data

$C_{18}H_{13}NO_3S$
M_r = 323.35
Monoclinic, $C2/c$
a = 18.469 (9) Å
b = 8.392 (6) Å
c = 19.240 (12) Å
β = 93.58 (4)°
V = 2976 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 296 K
0.50 × 0.50 × 0.20 mm

Data collection

Rigaku AFC-6*S* diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.893, T_{max} = 0.955
3419 measured reflections
3419 independent reflections
1307 reflections with $I > 2\sigma(I)$
3 standard reflections
every 150 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.191
S = 1.08
3419 reflections
214 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

The H atoms were included in the riding-model approximation, with C—H = 0.93 Å, and with U_{iso}(H) = 1.15–1.20U_{eq}(C).

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); 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*.

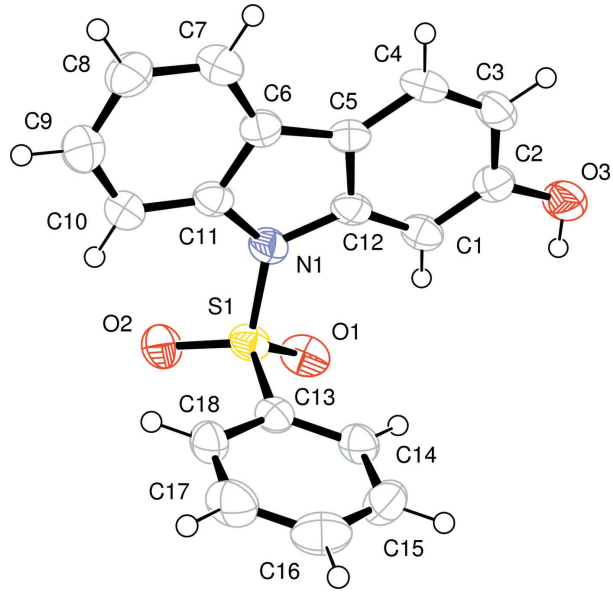


Figure 1
The molecular structure of (I), showing the atom-labelling scheme and with 50% probability displacement ellipsoids.

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