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Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: AB1422). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-Phenylsulfonyl-3-methylbenz[f]indole

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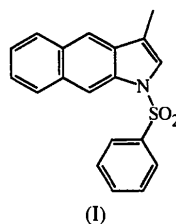
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Abstract

In the title compound, C₁₉H₁₅NO₂S, the benz[f]indole ring system is essentially planar, with maximum and mean deviations for the 13 ring atoms of 0.051 (2) and 0.023 (4) Å. The N atom lies 0.199 (1) Å out of the plane of the three atoms bonded to it. The phenyl group forms a dihedral angle of 82.83 (5)° with the benz[f]indole plane. The S—N distance is 1.646 (1) Å and the S=O distances average 1.426 (1) Å.

Comment

Benz[f]tryptophan is of interest as a hybrid intrinsic/extrinsic fluorescence probe of peptide structure and dynamics. Tryptophan is a very useful intrinsic fluorescence probe and the additional benzannulation shifts the absorbance and emission to the red of tryptophan. In theory, benz[f]tryptophan could replace tryptophan in peptide hormones and retain the same or similar biological activity, while giving a unique fluorescence emission even in the presence of a protein receptor. The title molecule, (I), was synthesized as an intermediate to benz[f]tryptophan and has much greater oxidative stability compared with 2-trimethylsilyl-3-methylbenz[f]indole, which air oxidizes during recrystallization to form the hydroperoxide (Morales & Fronczek, 1996).



The N atom is slightly non-planar and lies 0.199 (1) Å out of the plane defined by the S, C1 and C4 atoms. This slight pyramidalization is similar to that seen in 1-phenylsulfonylindole [0.179 (1) Å] and similar compounds (Beddoes *et al.*, 1986). The conformation of the N-phenylsulfonyl group with respect to the ring system is described by torsion angles O1—S—N1—C4 of −54.5 (1)° and O1—S—C14—C19 of 19.2 (2)°, which cause the best planes of the benz[f]indole and phenyl rings to form a dihedral angle of 82.83 (5)°. The corresponding angles in 1-phenylsulfonylindole are −38.3,

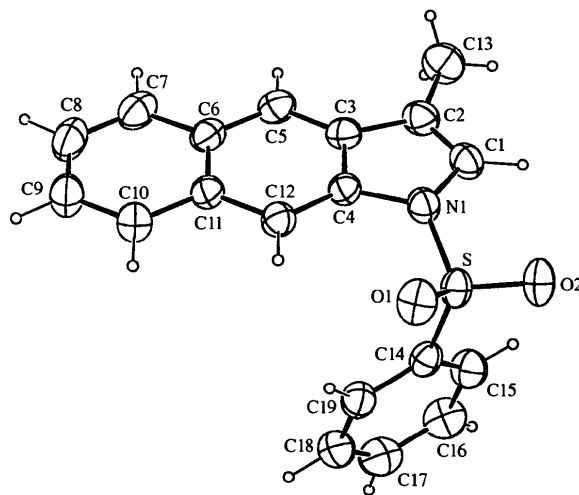


Fig. 1. The title molecule shown with ellipsoids at the 40% probability level and H atoms represented with arbitrary radii.

8.9 and 91.72°. Beddoes *et al.* (1986) have discussed the conformations of arylsulfonamides and have found these to be fairly typical values.

A search of the CSD (Allen, Kennard & Taylor, 1983) for the benz[*f*]indole substructure found in the present compound yielded no hits, except for three benzocarbazoles, which have a benzo group fused at C1—C2 (Drager, Haber, Erfanian-Abdoust, Pindur & Sattler, 1994; Seetharaman & Rajan, 1995*a,b*).

Experimental

The title compound was prepared by a Pd-catalyzed cyclization between 1-trimethylsilylpropyne and 3-bromo-2-aminonaphthalene, followed by *in situ* treatment with HCl gas to remove the trimethylsilyl group, and treatment with lithium diisopropylamide and benzenesulfonyl chloride to generate the title molecule. Crystals were grown by slow evaporation of a hexanes-dichloromethane mixture.

Crystal data

C₁₉H₁₅NO₂S
M_r = 321.40
 Triclinic
 P $\bar{1}$
a = 8.1524 (8) Å
b = 9.3328 (4) Å
c = 11.4597 (6) Å
 α = 66.924 (4)°
 β = 78.185 (6)°
 γ = 83.396 (5)°
V = 784.56 (6) Å³
Z = 2
D_x = 1.360 Mg m⁻³
D_m not measured

Cu K α radiation
 λ = 1.54184 Å
 Cell parameters from 25 reflections
 θ = 16–43°
 μ = 1.858 mm⁻¹
T = 293 K
 Prism
 0.38 × 0.37 × 0.12 mm
 Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
 T_{\min} = 0.609, T_{\max} = 0.799
 3486 measured reflections
 3246 independent reflections

3135 reflections with $I > \sigma(I)$
 R_{int} = 0.010
 θ_{max} = 74.88°
 h = 0 → 10
 k = -11 → 11
 l = -13 → 14
 3 standard reflections
 frequency: 120 min
 intensity decay: -0.27%

Refinement

Refinement on F^2
 $R(F)$ = 0.037
 $wR(F^2)$ = 0.056
 S = 1.572
 3135 reflections
 269 parameters
 H atoms refined isotropically
 $w = 4F_o^2/[\sigma^2(F_o^2) + 0.0004F_o^4]$
 $(\Delta/\sigma)_{\text{max}}$ = 0.022

$\Delta\rho_{\text{max}}$ = 0.22 e Å⁻³
 $\Delta\rho_{\text{min}}$ = -0.30 e Å⁻³
 Extinction correction: isotropic (Zachariasen, 1963)
 Extinction coefficient: 1.87 (3) × 10⁻⁵
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. Selected geometric parameters (Å, °)

S—O1	1.425 (1)	C3—C5	1.377 (2)
S—O2	1.428 (1)	C4—C12	1.371 (2)
S—N1	1.646 (1)	C5—C6	1.405 (2)
S—C14	1.758 (1)	C6—C7	1.425 (2)
N1—C1	1.422 (2)	C6—C11	1.427 (2)
N1—C4	1.418 (2)	C7—C8	1.359 (3)
C1—C2	1.335 (2)	C8—C9	1.400 (3)
C2—C3	1.447 (2)	C9—C10	1.367 (2)
C2—C13	1.499 (3)	C10—C11	1.418 (2)
C3—C4	1.420 (2)	C11—C12	1.415 (2)
O1—S—O2	120.20 (6)	N1—C1—C2	110.3 (1)
O1—S—N1	107.36 (6)	C1—C2—C3	107.9 (1)
O1—S—C14	108.54 (7)	C1—C2—C13	126.8 (1)
O2—S—N1	105.83 (7)	C3—C2—C13	125.3 (1)
O2—S—C14	108.89 (6)	C2—C3—C4	107.7 (1)
N1—S—C14	104.99 (6)	C2—C3—C5	133.1 (1)
S—N1—C1	122.71 (8)	N1—C4—C3	106.4 (1)
S—N1—C4	124.4 (1)	N1—C4—C12	130.6 (1)
C1—N1—C4	107.6 (1)		

C—H distances are in the range 0.90 (2)–1.00 (2) Å, while B_{iso} values for H atoms are in the range 4.0 (3)–9.1 (6) Å².

Data collection: CAD-4 Operations Manual (Enraf–Nonius, 1977). Cell refinement: CAD-4 Operations Manual. Data reduction: MolEN PROCESS (Fair, 1990). Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990) (direct methods). Program(s) used to refine structure: MolEN LSFM. Molecular graphics: ORTEPII (Johnson, 1976). Software used to prepare material for publication: MolEN CIF IN.

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