

The fused heterocycle 2-benzyl-3-methyl-1-phenyl-4-phenylsulfonyl-2*H*,4*H*-pyrrolo[3,4-*b*]indole

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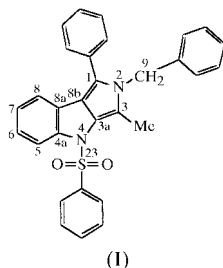
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Crystal structure determination of the title molecule, C₃₀H₂₄N₂O₂S, reveals that the pyrrole ring in this fused heterocycle, although presumably strained and reactive in cycloaddition reactions, does not differ appreciably from *N*-methylpyrrole except for a shorter C—C single bond [1.409 (4) Å] in the pyrrole ring.

Comment

Fused heterocycles such as the title compound, (I), are finding significant use as stable synthetic analogs of indole-2,3-quinodimethane for the construction of carbazoles and related natural products (Sha, 1996; Daly *et al.*, 1997). In continuation of our interest in these ring systems (Pelkey & Gribble, 1997; Gribble *et al.*, 1998), we needed to confirm the structure of the major product formed in the reaction between the mesoionic münchnone 3-benzyl-2-methyl-4-phenyl-1,3-oxazolium-5-olate and 3-nitro-1-(phenylsulfonyl)indole to give either 2-benzyl-3-methyl-1-phenyl-4-phenylsulfonyl-2*H*,4*H*-pyrrolo[3,4-*b*]indole, (I), or the isomeric 2-benzyl-1-methyl-3-phenyl-4-phenylsulfonyl-2*H*,4*H*-pyrrolo[3,4-*b*]indole, in which the methyl and phenyl groups are interchanged. This novel chemistry has been published for symmetrical münchnones, which give unambiguous results (Gribble *et al.*, 1998).



The crystal structure determination reported herein supports our tentative isomer assignment using NMR methods that (I) is the major product of this 1,3-dipolar cycloaddition

reaction. The present paper appears to report the first crystal structure of the pyrrolo[3,4-*b*]indole ring system. The tricyclic fused ring system is essentially planar as anticipated. The torsion angle at C1 with the phenyl ring, N2—C1—C11—C16, is 52.2 (4)°, unlike biphenyl which is coplanar (Trotter, 1961). The dihedral angle between the least squares planes of the *N*-benzyl phenyl ring, C17—C22, and the pyrrole ring is 96.9 (2)°, while that between the phenylsulfonyl ring, C26—C31, and the pyrroloindole ring system, C1/N2/C3/C3a/N4/C4a/C8a/C8b, is 60.3 (2)°. These conformations are more or less anticipated, since π -conjugation between these phenyl rings and the pyrroloindole ring system is non-existent.

The pyrrole C=C double bonds [C1=C8b 1.378 (4) and C3=C3a 1.367 (4) Å] are slightly shorter than the comparable bond length in *N*-benzylisindole (1.384 Å; Bonnett *et al.*, 1985). Likewise, the pyrrole ring N—C single bonds [C1—N2 1.393 (4) and N2—C3 1.404 (3) Å] are slightly longer than the comparable bond in *N*-benzylisindole (1.365 Å; Bonnett *et al.*, 1985). This indicates that the pyrrole ring in (I) is more 'pyrrole-like' and less delocalized than the pyrrole ring in *N*-benzylisindole. For comparison, the C=C double bond in *N*-methylpyrrole is 1.35 Å and the N—C bond length is 1.40 Å, as determined by electron diffraction (Vilkov *et al.*, 1962). The single bond between the two five-membered rings [C3a—C8b 1.409 (4) Å] is slightly compressed compared with the same (C3—C4) bond in *N*-methylpyrrole (1.43 Å) and *N*-benzylisindole (1.429 Å), as a consequence of the two fused aromatic five-membered rings.

The mean deviation of the *N*-benzyl pyrrole nitrogen, N2, from the plane of its nearest three neighbors, C1, C3 and C9, is −0.082 (8) Å, while for the *N*-phenylsulfonyl nitrogen, N4, and its nearest three neighbors, C3a, C4a and S23, it is −0.437 (1) Å, making N4 significantly pyramidal. The internal bond angles in the *N*-benzylpyrrole ring are very similar to those reported for *N*-benzylisindole.

In summary, the *N*-benzyl pyrrole ring, which is embedded in the highly congested title pyrroloindole, (I), is structurally

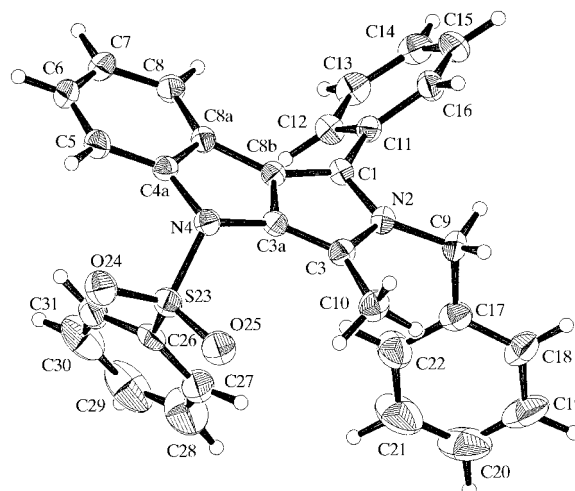


Figure 1
ORTEPII (Johnson, 1976) diagram of (I) showing 30% probability displacement ellipsoids. H atoms are drawn as spheres of arbitrary radii.

similar to a normal pyrrole ring, although it is certainly more strained than a simple pyrrole by virtue of being fused to another five-membered ring at the C3a and C8b positions.

Experimental

Compound (I) was prepared by our general method from 3-nitro-1-phenylsulfonylindole and *N*-benzyl-*N*-benzoylalanine (Gribble *et al.*, 1998), and recrystallized from dichloromethane to give light yellow prisms (m.p. 444–445 K). The full synthetic details will be described separately.

Crystal data

| | |
|--------------------------------|--|
| $C_{30}H_{24}N_2O_2S$ | $Z = 2$ |
| $M_r = 476.59$ | $D_x = 1.293 \text{ Mg m}^{-3}$ |
| Triclinic, $P\bar{1}$ | Mo $K\alpha$ radiation |
| $a = 10.300 (1) \text{ \AA}$ | Cell parameters from 20 reflections |
| $b = 13.390 (1) \text{ \AA}$ | $\theta = 6.2\text{--}7.6^\circ$ |
| $c = 9.322 (7) \text{ \AA}$ | $\mu = 0.163 \text{ mm}^{-1}$ |
| $\alpha = 105.65 (9)^\circ$ | $T = 296 \text{ K}$ |
| $\beta = 98.62 (9)^\circ$ | Prism, light yellow |
| $\gamma = 88.45 (3)^\circ$ | $0.5 \times 0.4 \times 0.4 \text{ mm}$ |
| $V = 1223.9 (4) \text{ \AA}^3$ | |

Data collection

| | |
|--|---------------------------|
| Rigaku AFC-6S diffractometer | $h = 0 \rightarrow 13$ |
| $\omega/2\theta$ scans | $k = -17 \rightarrow 17$ |
| 5951 measured reflections | $l = -12 \rightarrow 11$ |
| 5637 independent reflections | 3 standard reflections |
| 2632 reflections with $I > 3\sigma(I)$ | every 150 reflections |
| $R_{\text{int}} = 0.026$ | intensity decay: -3.0% |
| $\theta_{\text{max}} = 27.50^\circ$ | |

Refinement

| | |
|--|--|
| Refinement on F | $(\Delta/\sigma)_{\text{max}} = 0.030$ |
| $R = 0.050$ | $\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$ |
| $wR = 0.196$ | $\Delta\rho_{\text{min}} = -0.36 \text{ e \AA}^{-3}$ |
| $S = 1.050$ | Extinction correction: |
| 5637 reflections | Zachariasen (1967) |
| 317 parameters | Extinction coefficient: |
| H-atom parameters not refined | $4.5 (2) \times 10^{-6}$ |
| $w = 1/[\sigma^2(F_o) + 0.00004 F_o ^2]$ | |

Idealized C–H distances are in the range 0.95–0.97 Å.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997–1999); program(s) used to solve structure: *SIR93* (Altomare *et al.*, 1993) and *DIRDIF94* (Beurskens *et al.*, 1994); program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993); software used to prepare material for publication: *TEXSAN for Windows*.

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Table 1

Selected geometric parameters (Å, °).

| | | | |
|----------|-----------|------------|-----------|
| S23–N4 | 1.674 (2) | C1–C8b | 1.378 (4) |
| N2–C1 | 1.393 (4) | C3–C3a | 1.367 (4) |
| N2–C3 | 1.404 (3) | C3a–C8b | 1.409 (4) |
| N2–C9 | 1.455 (4) | C4a–C8a | 1.412 (4) |
| N4–C3a | 1.449 (3) | C8a–C8b | 1.454 (4) |
| N4–C4a | 1.458 (4) | | |
| C1–N2–C3 | 111.3 (2) | S23–N4–C3a | 116.4 (2) |
| C1–N2–C9 | 125.2 (2) | S23–N4–C4a | 115.8 (2) |
| C3–N2–C9 | 122.5 (2) | C3a–N4–C4a | 104.0 (2) |

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1236). Services for accessing these data are described at the back of the journal.

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