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4-Ethyl-8-methyl-6-phenyl-1-(phenylsulfonyl)-1*H*,6*H*-pyrrolo[3,4-*f*]indole-5,7(1*H*,6*H*)-dione

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Key indicators

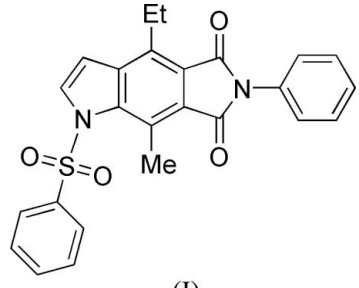
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.013 Å
R factor = 0.055
wR factor = 0.204
Data-to-parameter ratio = 12.4

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

The title molecule, $C_{25}H_{20}N_2O_4S$, is a rare example of the 1*H*,6*H*-pyrrolo[3,4-*f*]indole ring system, and shows the tricyclic fused ring system to be essentially planar.

Comment

The synthesis of complex indole ring systems continues to be an important goal in heterocyclic chemistry (Gribble, 2000). One synthetic approach to such fused indoles is *via* the Diels–Alder cycloaddition of furo[3,4-*b*]pyrroles with dienophiles followed by loss of water from the cycloadduct. We recently described the first generation of the furo[3,4-*b*]pyrrole ring system and demonstrated its utility in this new indole ring synthesis (Moskalev & Gribble, 2002). As this heterocycle, 4-ethyl-6-methyl-1-(phenylsulfonyl)-1*H*-furo[3,4-*b*]pyrrole, was too unstable for X-ray crystallographic analysis, the title *N*-phenylmaleimide Diels–Alder product, (I), was investigated (Fig. 1).



(I)

The tricyclic fused ring system is essentially planar, as anticipated. The mean deviation from the tricyclic pyrroloindole plane is 0.024 (1) Å. The *N*-phenyl ring defined by C21–C26 is twisted out of the plane of the tricyclic ring system with a dihedral angle of 40.9 (3)°. The dihedral angle between the least-squares plane of the pyrroloindole and the phenylsulfonyl benzene ring is 80.5 (4)°, indicating a greater degree of twisting over that normally seen in related ring systems (Simon *et al.*, 2000), perhaps due to a repulsive interaction with the methyl group. This repulsion is further revealed by the larger C11—N1—S1 bond angle, 132.0 (4)°, compared with the C2—N1—S1 bond angle of 119.9 (4)°. The sum of the angles around the indole atom N1 is 359.3°, indicating sp^2-hybridization, as does the sum of the angles around the pyrrole atom N2 (360.0°). The geometry about the sulfonyl group is comparable to that observed in other *N*-(phenylsulfonyl)indoles (Beddoes *et al.*, 1986; Yokum & Fronczek, 1997; Simon *et al.*, 2000; Seshadri *et al.*, 2002; Sonar *et al.*, 2004).

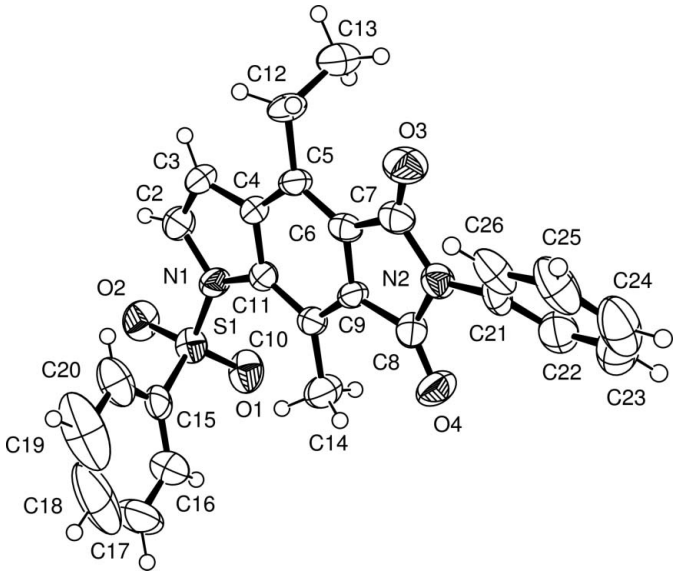


Figure 1
Molecular structure of (I), showing the atom labeling and 50% probability displacement ellipsoids.

Experimental

Compound (I) was prepared according to our published procedure (Moskalev & Gribble, 2002). Crystallization from ethanol gave colorless crystals suitable for X-ray analysis; m.p. 431–433 K.

Crystal data

$C_{25}H_{20}N_2O_4S$
M_r = 444.49
Monoclinic, $C2/c$
a = 19.751 (4) Å
b = 12.534 (5) Å
c = 17.747 (4) Å
β = 105.820 (17)°
V = 4227 (2) Å^3
Z = 8
D_x = 1.397 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 296 (2) K
Prism, colorless
0.50 × 0.30 × 0.20 mm

Data collection

Rigaku AFC-6S diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.911, T_{max} = 0.963
3606 measured reflections
3606 independent reflections
1135 reflections with $I > 2\sigma(I)$
θ_{max} = 27.5°
3 standard reflections
every 150 reflections
intensity decay: 0.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.204
S = 1.0
3606 reflections
291 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0594P)^2 + 10.5049P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = **0.00o**
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00068 (18)

The H atoms were included in the riding model approximation with C—H = 0.93-0.97 Å, and with U_{iso}(H) = 1.18–1.20U_{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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