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(*Z*)-2-[1-(4-Methylphenylsulfonyl)-1*H*-indol-3-ylmethylene]-1-azabicyclo[2.2.2]-octan-3-one

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Key indicators

Single-crystal X-ray study
T = 90 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.036
wR factor = 0.095
Data-to-parameter ratio = 16.7

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

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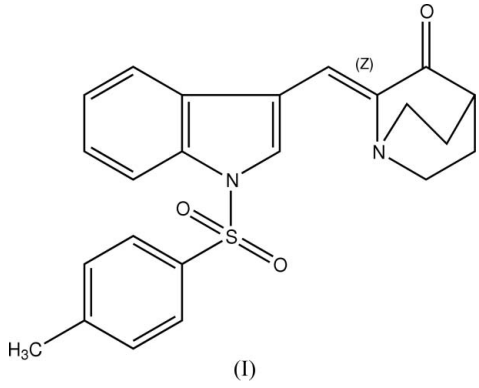
In the title compound, $C_{23}H_{22}N_2O_3S$, the indole ring system is planar and makes a dihedral angle with the 4-methylbenzene ring of 83.37 (3)°. The double bond connecting the azabicyclic and indole groups adopts a *Z* geometry. The H atoms of the 4-methyl group are disordered.

Comment

A single isomer of the title compound, (I), was prepared by base-catalysed condensation, as part of our continuing work on the structure and conformation of biologically active indole analogues (Sonar *et al.*, 2004). In order to confirm the double-bond geometry of this compound, its X-ray crystal structure determination has been carried out.



(I)

The molecular structure and atom-numbering scheme of (I) are shown in Fig. 1. Selected geometric parameters are presented in Table 1. The indole ring system is planar with bond distances and angles comparable with those previously reported for other indole derivatives (Mason *et al.*, 2003). The angles around the S atom are distorted from the ideal tetrahedral values (Table 1), with largest deviations found for O2—S1—O1, O2—S1—N1, and O1—S1—N. This observation is in agreement with previously reported values for the sulfonyl group, and is due to the repulsive interaction between the short S═O bonds (Seshadri *et al.*, 2002). The S—O, S—C, and S—N distances are comparable with those found in *N*-phenylsulfonamides (Gomes *et al.*, 1993). The conformation of the 4-methylbenzenesulfonyl group with respect to the indole ring system is described by the torsion angles O1—S1—N1—C2, O2—S1—N1—C9 and N1—S1—C19—C20 (Table 1). The 4-methylbenzene ring linked to the sulfonyl group is orthogonal to the indole ring system, forming a dihedral angle of 83.37 (3)°.

In the molecule of (I), the C11—C18 bond is in a *trans* disposition with respect to the C3—C10 bond. The double bond has a nearly planar arrangement. Deviations from ideal

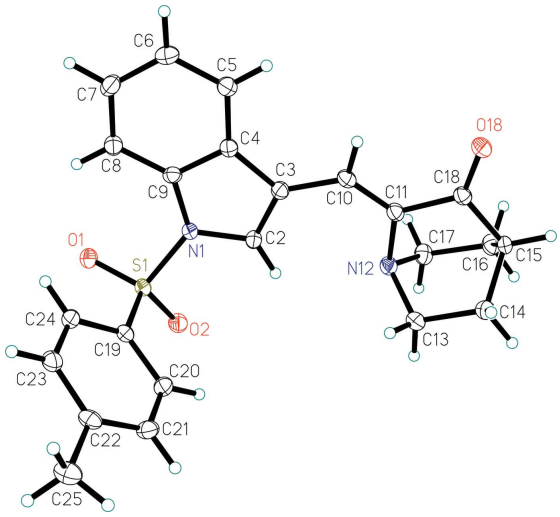


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

geometry are observed in the bond angles around atoms C3, C10 and C11. The bond angles C2—C3—C10, C3—C10—C11, and N12—C11—C18 are distorted because of the strain induced by the double-bond linkage at C10—C11. Within the azabicyclic system, very small distortions are observed. The small C2—C3—C10—C11 torsion angle indicates that there is only a small deviation of the indole ring system from the plane of the double bond connected to the azabicyclic system. The C3—C10 bond length, when compared with the standard value for a single bond connecting a C_{ar} atom to a Csp^2 atom [1.470 (15) Å; Allen *et al.*, 1987], suggests a weak conjugation, beginning at atom O18 and extending through to the aromatic ring, which is also evident from the bond lengths C11—C18 and C18—O18.

Experimental

Compound (I) was prepared by base-catalyzed condensation following the method described previously for the benzenesulfonyl analogue (Sonar *et al.*, 2004), but utilizing 1-(4-methylbenzenesulfonyl)-1*H*-indole-3-carboxaldehyde instead of 1-benzenesulfonyl-1*H*-indole-3-carboxaldehyde. ^{1}H NMR ($CDCl_3$): δ 1.94 (*td*, 4H), 2.24 (*s*, 3H), 2.54 (*p*, 1H), 2.84–2.93 (*m*, 2H), 3.08–3.17 (*m*, 2H), 7.13 (*s*, 2H), 7.16 (*d*, 2H), 7.18–7.26 (*m*, 1H), 7.63 *d*, 1H), 7.69 (*d*, 2H), 7.86 (*d*, 1H), 8.58 (*s*, 1H). ^{13}C NMR ($CDCl_3$): δ 22.0, 26.4, 40.7, 47.5, 113.7, 115.1, 115.9, 119.4, 123.9, 125.2, 127.0, 130.1, 130.5, 130.6, 134.5, 135.1, 144.8, 145.4, 205.3.

Crystal data

$C_{23}H_{22}N_2O_3S$
M_r = 406.49
Triclinic, $P\overline{1}$
a = 7.9050 (1) Å
b = 10.6596 (2) Å
c = 12.2642 (2) Å
α = 93.2146 (7)°
β = 101.1313 (8)°
γ = 107.3499 (7)°
V = 960.70 (3) Å^3
Z = 2
D_x = 1.405 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4384 reflections
θ = 1.0–27.5°
μ = 0.20 mm^{-1}
T = 90.0 (2) K
Block, yellow
0.25 × 0.25 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
ω scans at fixed χ = 55°
Absorption correction: multi-scan (*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.952, T_{max} = 0.962
8764 measured reflections
4419 independent reflections
3686 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.095
S = 1.06
4419 reflections
264 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0436P)^2 + 0.4054P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.010
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C2	1.3965 (17)	C10—C11	1.3388 (19)
N1—S1	1.6646 (11)	C11—C18	1.4901 (18)
C2—C3	1.3643 (19)	O18—C18	1.2204 (16)
C3—C10	1.4474 (19)	S1—O1	1.4276 (10)
C2—C3—C10	128.47 (12)	O1—S1—N1	106.10 (6)
C10—C3—C4	124.39 (12)	O2—S1—N1	105.82 (6)
C11—C10—C3	128.49 (13)	N1—S1—C19	105.73 (6)
C10—C11—C18	122.02 (12)	O18—C18—C11	125.05 (13)
N12—C11—C18	113.82 (11)	C11—C18—C15	110.24 (11)
O1—S1—O2	121.41 (6)		
C2—C3—C10—C11	5.6 (2)	C10—C11—C18—O18	−6.9 (2)
C2—N1—S1—O1	−162.15 (11)	N1—S1—C19—C20	−91.68 (12)
C2—N1—S1—O2	−31.94 (13)		

All H atoms were located in difference Fourier syntheses and were subsequently positioned geometrically and refined with a riding model. Bond distances to parent atoms were set at 0.95 (C_{ar}—H), 0.98 (C_{Me}—H), 0.99 (C_{sec}—H), and 1.00 Å (C_{tert}—H) and U_{iso}(H) = 1.2 or 1.5 times U_{eq}(C). The methyl H atoms were disordered over two sites using a riding model defined by the command AFIX 127 in *SHELXL97* (Sheldrick, 1997).

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1995); software used to prepare material for publication: *SHELXL97* and local procedures.

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Gomes, A. C., Biswas, G., Biswas, S., Biswas, G. K., Iitaka, Y. & Bannerji, A. (1993). *J. Crystallogr. Spectrosc. Res.* **23**, 513–517.
Mason, M. R., Barnard, T. S., Segla, M. F., Xie, B. & Kirschbaum, K. (2003). *J. Chem. Crystallogr.* **33**, 531–540.
Nonius (1999). *COLLECT*. Nonius, BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Seshadri, P. R., Velmurugan, D., Govindaraj, J., Kannadasan, S., Srinivasan, P. C., Shanmuga Sundara Raj, S., Fun, H.-K. & Kim, M. J. (2002). *Acta Cryst.* C**58**, o700–o703.
Sheldrick (1995). *XP* in *SHELXTL/PC*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sonar, V. N., Parkin, S. & Crooks, P. A. (2004). *Acta Cryst.* C**60**, o659–o661.