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1′-Methyl-4′-tolyl-1*H*-indole-3-spiro-2′-pyrrolidine-3′-spiro-5″-(thiazolo[3,2-*b*]-[1,2,4]triazole)-2,6″(3*H*,5″*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.066
wR factor = 0.156
Data-to-parameter ratio = 13.1

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

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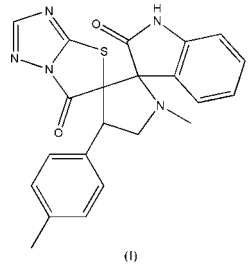
The title compound, $C_{22}H_{19}N_5O_2S$, was synthesized by an intermolecular [3 + 2]-cycloaddition of the azomethine ylide derived from isatin and sarcosine by a decarboxylative route and 5-(4-methylbenzylidene)thiazolo[3,2-*b*][1,2,4]triazol-6-one. In the molecule, the two spiro junctions link a planar 2-oxindole ring, a pyrrolidine ring in an envelope conformation and a thiazolo[3,2-*b*][1,2,4]triazol-6-one system.

Comment

Spiro-compounds represent an important class of naturally occurring substances characterized by highly pronounced biological properties (Kobayashi *et al.*, 1991; James *et al.*, 1991). 1,3-Dipolar cycloaddition reactions are important processes for the construction of spiro-compounds (Caramella & Grunanger, 1984).



(I)

The title compound, (I), was synthesized by an intermolecular [3 + 2]-cycloaddition of the azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 5-(4-methylbenzylidene)thiazolo[3,2-*b*][1,2,4]triazol-6-one. The molecular structure of (I) is illustrated in Fig. 1. In the molecule, the two spiro junctions link a planar 2-oxindole ring, a pyrrolidine ring in an envelope conformation and a thiazolo[3,2-*b*][1,2,4]triazol-6-one ring.

Experimental

A mixture of 5-(4-methylbenzylidene)thiazolo[3,2-*b*][1,2,4]triazol-6-one (1 mmol), isatin (1 mmol) and sarcosine (1 mmol) was refluxed in methanol (60 ml) until the starting material had disappeared, as confirmed by thin-layer chromatography. When the reaction was complete, the solvent was removed *in vacuo* and the residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate = 2:1), giving the title compound, (I) (m.p. 480–481 K); IR (KBr): 3225.6 (NH), 1766.2, 1713.1 (C═O) cm^{-1}; ^1H NMR (p.p.m.): 2.31 (*s*, 3H, N—CH_3), 3.64 (*m*, 1H, —CH_2), 4.24 (*m*, 1H, —CH_2), 4.67 (*m*, 1H, —CH), 6.79–7.79 (*m*, 9H, Ar—H), 7.85 (*bs*, 1H, —NH). 20 mg of (I) was dissolved in 15 ml of dioxane. The solution was kept at room temperature for 15 d and natural evaporation gave colorless single crystals of (I) suitable for X-ray analysis.

Crystal data

$C_{22}H_{19}N_5O_2S$	D_x = 1.363 Mg m^{-3}
M_r = 417.48	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1005 reflections
a = 19.153 (6) Å	θ = 2.6–24.4°
b = 6.2858 (19) Å	μ = 0.19 mm^{-1}
c = 16.968 (5) Å	T = 293 (2) K
β = 95.218 (5)°	Block, colorless
V = 2034.3 (11) Å^3	0.22 × 0.08 × 0.06 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3582 independent reflections
φ and ω scans	2218 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	R_{int} = 0.064
T_{min} = 0.824, T_{max} = 0.990	θ_{max} = 25.0°
9314 measured reflections	$h = -16 \rightarrow 22$
	$k = -7 \rightarrow 4$
	$l = -20 \rightarrow 19$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.066	$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.156	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.04	$\Delta\rho_{max}$ = 0.75 e Å^{-3}
3582 reflections	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
273 parameters	

H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(carrier). The largest peak in the difference Fourier map is situated at 1.98 Å from atom C4.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

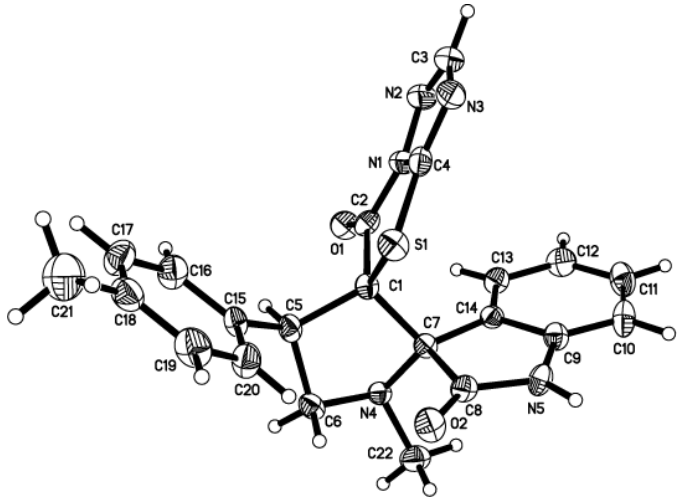


Figure 1
The molecular structure of (I), drawn with 30% probability displacement ellipsoids.

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