Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1′-Methyl-4′-(1-naphthyl)-1*H*-indole-3-spiro-2′-pyrrolidine-3′-spiro-2″-thiazolo[3,2-*a*]-benzimidazole-2(3*H*),3″(2″*H*)-dione

Xiu-Ling Zhang,[a]* Ai-Zhen Liu[a] and Mei-Fang Jin[b]

[a]Chemistry Department, Dezhou University, Dezhou Shandong 253011, People's Republic of China, and [b]Chemical Engineering Department, Anyang Institute of Technology, Anyang Henan 455000, People's Republic of China

Correspondence e-mail: zxl1962432@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.114
Data-to-parameter ratio = 12.9

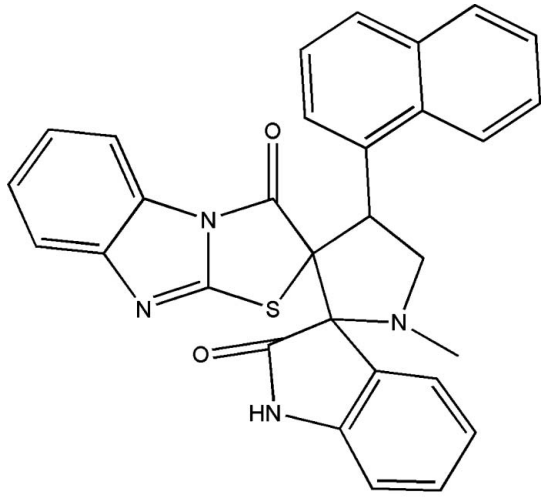
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 9 May 2006
Accepted 14 June 2006

In the title compound, $C_{30}H_{22}N_4O_2S$, two spiro junctions link a planar 2-oxoindole ring, a pyrrolidine ring and a thiazolo[3,2-*a*][1,3]benzimidazol-3-one system. Molecules form dimers connected by N—H···N hydrogen bonds.

Comment

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



(I)

In this paper, the synthesis and structure of the title two-spiro-junction compound, (I) (Fig. 1), are reported. Two spiro junctions exist in the molecule, which contains a 2-oxoindole ring, a pyrrolidine ring and a thiazolo[3,2-*a*][1,3]benzimidazol-3-one system. The 2-oxoindoline ring (C10–C17/N4) is essentially planar, with a mean deviation of 0.054 (5) Å. The packing can be described as a dimeric arrangement of molecules linked through N—H···N hydrogen bonds, as shown in Table 1. Atom N2 of the thiazolo[3,2-*a*][1,3]benzimidazol-3-one system acts as an acceptor in a hydrogen-bond interaction with the 2-oxoindole N4 atom in a symmetry-related molecule at (−*x*, -*y* + 1, -*z* + 1).

Experimental

A mixture of 2-(1-naphthylmethylene)thiazolo[3,2-a][1,3]benzimidazol-3-one (1 mmol), isatin (1 mmol) and sarcosine (1 mmol) was

refluxed in methanol (60 ml) until the disappearance of the starting material was evidenced by thin-layer chromatography. After evaporation of the solvent, the residue was separated by column chromatography (silica gel; petroleum ether–ethyl acetate 5:1) to give (I) (m.p. 507–508 K). Compound (I) (20 mg) was dissolved in acetone (15 ml) and the solution was left to evaporate at room temperature for 8 d, giving colorless single crystals of (I) suitable for X-ray analysis.

Crystal data

$C_{30}H_{22}N_4O_2S$	$Z = 8$
$M_r = 502.58$	$D_x = 1.358$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation
$a = 16.126$ (4) Å	$\mu = 0.17$ mm^{-1}
$b = 12.852$ (4) Å	$T = 294$ (2) K
$c = 23.725$ (6) Å	Block, colorless
$V = 4917$ (2) Å^3	0.24 × 0.20 × 0.10 mm

Data collection

Bruker APEX CCD area-detector diffractometer	24035 measured reflections
φ and ω scans	4331 independent reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	2725 reflections with $I > 2\sigma(I)$
$T_{min} = 0.961$, $T_{max} = 0.983$	$R_{int} = 0.071$
	$\theta_{max} = 25.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.4279P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.043$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.114$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
$S = 1.06$	$\Delta\rho_{min} = -0.23$ e Å^{-3}
4331 reflections	
335 parameters	
H-atom parameters constrained	

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···N2^{i}	0.86	2.14	2.994 (3)	174

Symmetry code: (i) $-x, -y+1, -z+1$.

H atoms were positioned geometrically and refined using a riding model [C—H = 0.93–0.98 Å and N—H = 0.83 Å, with U_{iso}(H) = 1.5U_{eq}(methyl C) and 1.2U_{eq}(C,N).

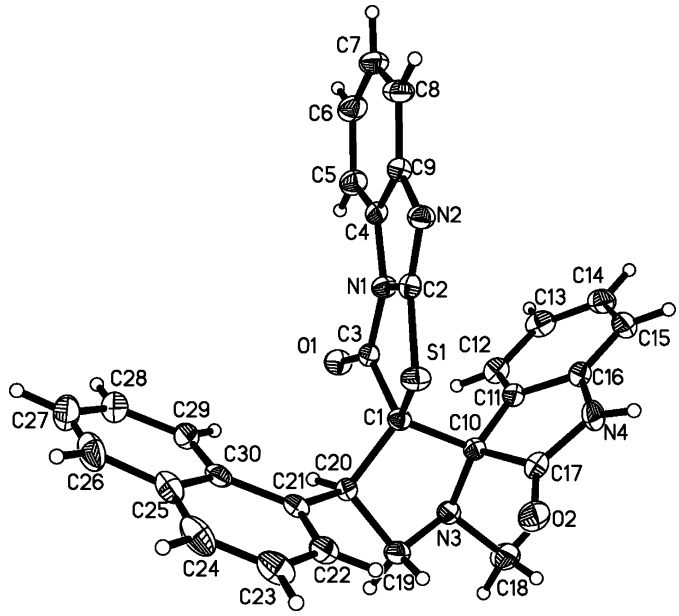


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids. H atoms are drawn as spheres of arbitrary radius.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

References

Bruker (1997). *SADABS*, *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Caramella, P. & Grunanger, P. (1984). *1,3-Dipolar Cycloaddition Chemistry*, edited by A. Padwa, Vol. 1, pp. 291–312. New York: Wiley.
James, D. M., Kunze, H. B. & Faulkner, D. J. (1991). *J. Nat. Prod.* **54**, 1137–1140.
Kobayashi, J., Tsuda, M., Agemi, K., Shigemori, H., Ishibashi, M., Sasaki, T. & Mikami, Y. (1991). *Tetrahedron*, **47**, 6617–6622.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.