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4′-(4-Methoxyphenyl)-1′-methyl-4″,5″,6″,7″-tetrahydro-1*H*-indole-3-spiro-2′-pyrrolidine-3′-spiro-2″-(thiazolo[3,2-*a*]pyrimidine)-2(3*H*),-3″(2″*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.048
wR factor = 0.113
Data-to-parameter ratio = 15.2

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

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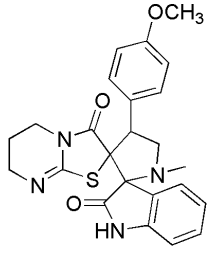
In the title compound, $C_{24}H_{24}N_4O_3S$, two spiro junctions link a planar 2-oxindole ring, a pyrrolidine ring in an envelope conformation and a 6,7-dihydro-5*H*-thiazolo[3,2-*a*]pyrimidin-3-one ring. Two molecules are connected into a centrosymmetric dimer by N—H···N hydrogen bonds.

Comment

Spiro-compounds represent an important class of naturally occurring substances, which in many cases exhibit important biological properties (Kobayashi *et al.*, 1991; James *et al.*, 1991). 1,3-Dipolar cycloaddition reactions are widely used for the construction of spiro-compounds (Caramella & Grunanger, 1984). In this paper, the structure of the title compound, (I), is reported. The compound was synthesized by the intermolecular [3 + 2]-cycloaddition of azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 2-(4-methoxybenzylidene)-6,7-dihydro-5*H*-thiazolo[3,2-*a*]pyrimidin-3-one (Mohan & Verma, 1993).



(I)

The molecular structure of (I) is shown in Fig. 1. There are two spiro junctions in the molecule which consists of a planar 2-oxindole ring, a pyrrolidine ring in an envelope conformation and a thiazolo[3,2-*a*]pyrimidine ring. Two molecules are linked into a centrosymmetric dimer by two N4—H···N2 hydrogen bonds, with an N···N distance of 2.887 (2) Å and an N—H···N angle 169°.

Experimental

A mixture of 2-(4-methoxybenzylidene)-6,7-dihydro-5*H*-thiazolo-[3,2-*a*]pyrimidin-3-one (1 mmol), isatin(1 mmol) and sarcosine (1 mmol) was refluxed in methanol (60 ml) until the disappearance of the starting material, as evidenced 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. 506 K); IR (KBr): 3351.4 (–NH), 1723.6, 1689.2 (C═O) cm^{-1}; ^1H NMR (δ, p.p.m.): 1.15 (*m*, 1H, $-CH_2$), 1.65 (*m*, 1H, $-CH_2$),

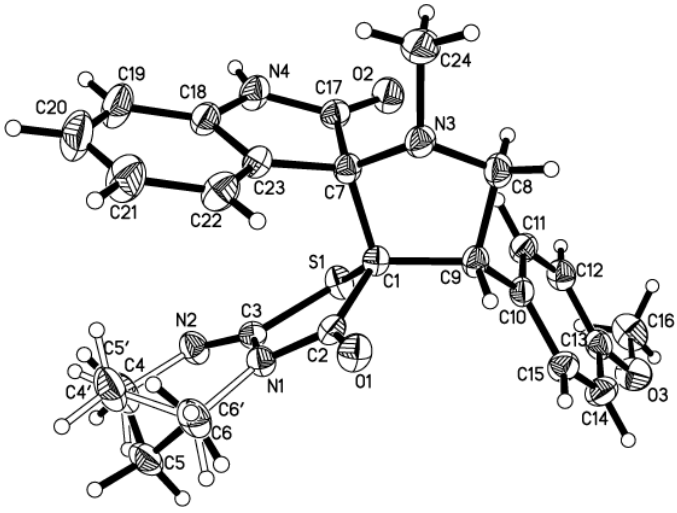


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids.

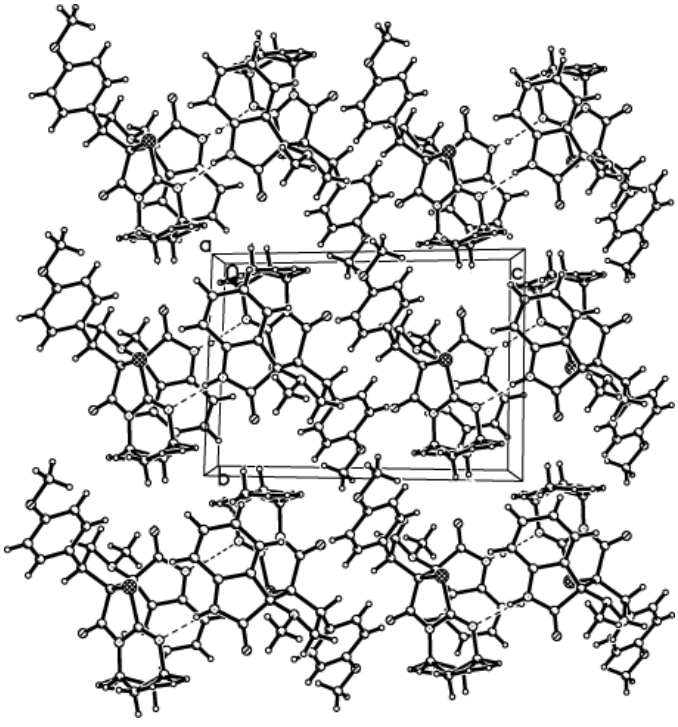


Figure 2
The crystal packing of (I), viewed along the *a* axis.

2.27(*s*, 3H, N—CH_3), 3.25 (*m*, 2H, CH_2), 3.37 (*m*, 2H, CH_2), 3.63 (*m*, 1H, —CH_2), 3.73 (*s*, 3H, —CH_3) 3.99 (*m*, 1H, —CH_2), 4.58 (*m*, 1H, —CH), 6.75–7.47 (*m*, 8H, Ar—H), 8.55 (*bs*, 1H, —NH). 20 mg of (I) were dissolved in 15 ml 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

$C_{24}H_{24}N_4O_3S$	$Z = 2$
$M_r = 448.53$	$D_x = 1.317$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 8.660$ (3) Å	Cell parameters from 692 reflections
$b = 9.843$ (4) Å	$\theta = 2.5$–24.7°
$c = 14.095$ (6) Å	$\mu = 0.18$ mm^{-1}
$\alpha = 88.374$ (7)°	$T = 293$ (2) K
$\beta = 73.054$ (6)°	Plate, colorless
$\gamma = 79.895$ (6)°	0.30 × 0.20 × 0.10 mm
$V = 1131.1$ (8) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	4585 independent reflections
φ and ω scans	3567 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	$R_{int} = 0.022$
$T_{min} = 0.861$, $T_{max} = 0.980$	$\theta_{max} = 26.4^\circ$
6236 measured reflections	$h = -10 \rightarrow 10$
	$k = -12 \rightarrow 10$
	$l = -10 \rightarrow 17$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.048$	$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.113$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.01$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
4585 reflections	$\Delta\rho_{min} = -0.29$ e Å^{-3}
301 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···N2^{i}	0.86	2.04	2.887 (4)	169

Symmetry code: (i) $-x, 1-y, 2-z$.

H atoms were positioned geometrically and treated in the riding-model approximation [C—H = 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$].

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*.

References

Bruker (1997). *SADABS, SMART, SAINT* and *SHELXTL*. Versions 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Caramella, P. & Grunanger, P. (1984). 1,3-*Dipolar Cycloaddition Chemistry*, Vol. 1, edited by A. Padwa, pp. 291–312. New York: Wiley.

James, D., Kunze, H. B. & Faulkner, D. (1991). *J. Nat. Prod*. **54**, 1137–1140.

Kobayashi, J., Tsuda, M., Agemi, K., Shigemori, H. Ishibashi, M., Sasaki, T. & Mikamiy, Y. (1991). *Tetrahedron*, **47**, 6617–6622.

Mohan, J. & Verma, P.(1993). *Indian J. Chem. Sect. B*, **32**, 986–988.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.