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Ethyl 4′-(4-methoxyphenyl)-1′,7″-dimethyl-2,3″-dioxo-5″-phenyl-2,3,2″,3″,4″,5″-tetrahydro-1*H*-indole-3-spiro-2′-pyrrolidine-3′-spiro-2″-(thiazolopyrimidine)-6″-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.053
wR factor = 0.137
Data-to-parameter ratio = 15.1

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

In the title compound, $C_{34}H_{32}N_4O_5S$, the two spiro junctions link a planar 2-oxindole ring, a pyrrolidine ring in an envelope conformation and a thiazolo[3,2-*a*]pyrimidine ring. Two molecules are connected by N—H···N hydrogen bonds, with an N···N distance of 3.027 (2) Å and an N—H···N angle of 140.2°.

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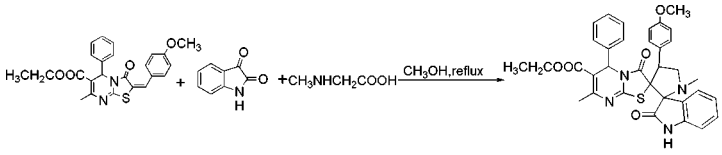
Comment

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)-7-methyl-3-oxo-5-phenyl-2,3-dihydro-5*H*-thiazolo[3,2-*a*]pyrimidine-6-carboxylic acid ethyl ester (Tozkoparan *et al.*, 1999).



(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 and a thiazolo[3,2-*a*]-rolidine ring (N3, C18, C17, C1 and C19) has an envelope conformation. Two molecules are connected by N—H···N hydrogen bonds, with an N···N distance of 3.027 (2) Å and an N—H···N angle 140.2°.

Experimental

A mixture of 2-(4-methoxybenzylidene)-7-methyl-3-oxo-5-phenyl-2,3-dihydro-5*H*-thiazolo[3,2-*a*]pyrimidine-6-carboxylic acid ethyl ester (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 = 5:1), giving the title compound (I) (m.p. 479–481 K); IR (KBr): 3351.4 (–NH), 1743.4, 1723.1, 1688.2 (C═O) cm^{-1}; ^1H NMR (δ, p.p.m.): 1.03 (*m*, 3H, $-CH_3$), 2.17 (*s*, 3H, $-CH_3$), 2.23 (*s*, 3H, N$-CH_3$), 3.45 (*m*, 1H, $-CH_2$), 3.73 (*s*, 3H, $-CH_3$), 3.99 (*m*, 1H, $-CH_2$), 4.03 (*m*, 2H, $-CH_2$), 4.57 (*m*, 1H, —CH), 5.72 (*s*, 1H, —CH),

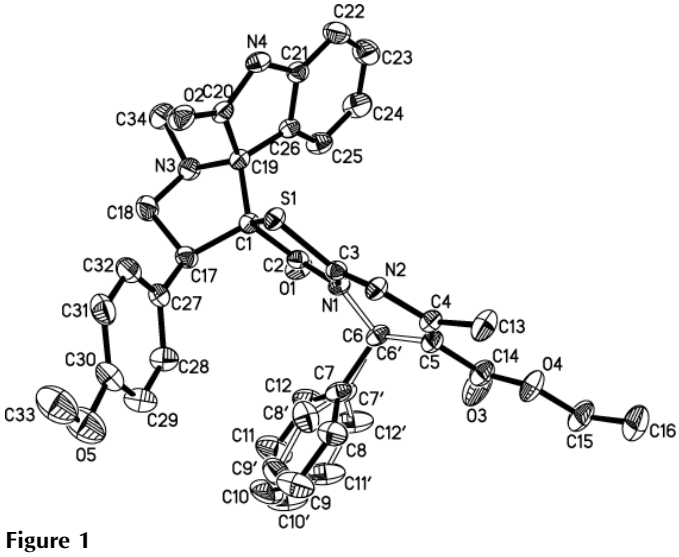


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids. H atoms have been omitted for clarity. The minor disorder component has primed atom labels.

6.69–7.74 (*m*, 13H, ArH), 7.86 (*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

$C_{34}H_{32}N_4O_5S$
M_r = 608.70
Monoclinic, $P2_1/n$
a = 14.990 (6) Å
b = 11.375 (5) Å
c = 17.815 (8) Å
β = 92.254 (6)°
V = 3035 (2) Å^3
Z = 4
D_x = 1.332 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 816 reflections
θ = 2.3–25.3°
μ = 0.16 mm^{-1}
T = 293 (2) K
Block, colorless
0.38 × 0.30 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.905, T_{max} = 0.960
14013 measured reflections
6236 independent reflections
3567 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 26.4°
$h = -18 \rightarrow 12$
$k = -14 \rightarrow 6$
$l = -21 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.137
S = 1.00
6236 reflections
413 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

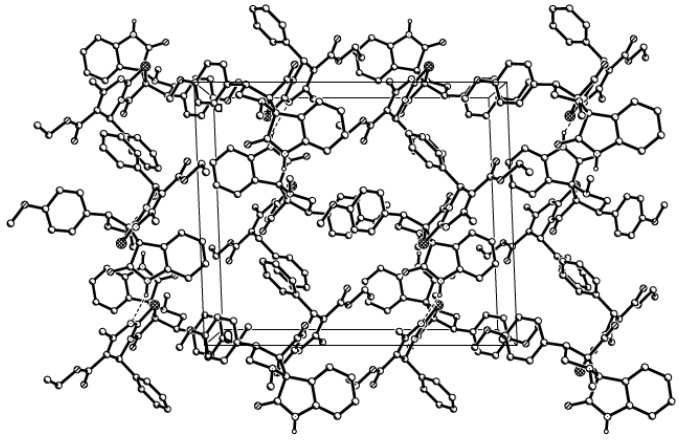


Figure 2
The crystal structure of (I), viewed along the *b* axis.

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···N2^{i}	0.86	2.31	3.027 (2)	140

Symmetry code: (i) $-\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

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)]. Atom C6 and the atoms of the attached phenyl ring are disordered over two sites. The ratio of site occupancies from the refinement was 0.64:0.36 (17).

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*.

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