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10″-(4-Methoxybenzylidene)-5″,4′-bis(4-methoxyphenyl)-1′-methyl-2,3,2″,3″,7″,8″,9″,10″-octahydro-1*H*,5″*H*,6″*H*-indole-3-spiro-2′-pyrrolidine-3′-spiro-2″-cyclohepteno[1,2-*d*]thiazolo[3,2-*a*]pyrimidine-2,3″-dione

Hong-Liang Chen, Ya-Qing Feng,* Xiao-Fen Hu and Xin-Gang Liu

School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: hlchen@tju.edu.cn

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.055
wR factor = 0.140
Data-to-parameter ratio = 14.8

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

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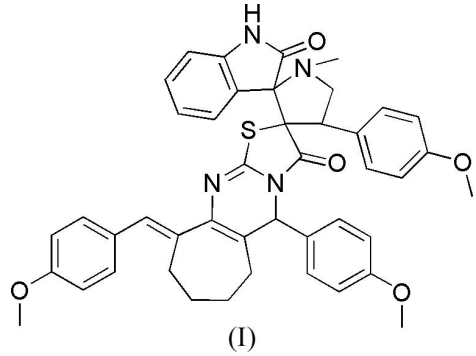
The title compound, $C_{44}H_{42}N_4O_5S$, was synthesized by the intermolecular [3+2] cycloaddition of an azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 5-(4-methoxyphenyl-10-(4-methoxy)benzylidene-2-(4-methoxy)benzylidene-2,3,6,7,8,9-hexahydro-5*H*,10*H*-cyclohepta[1,2-*d*]thiazolo[3,2-*a*]pyrimidin-3-one. In the molecule, the two spiro junctions link a planar 2-oxindole ring, a pyrrolidine ring in an envelope conformation and a 10-(arylmethylene)hexahydrocyclohepta[1,2-*d*]thiazolo-[3,2-*a*]pyrimidin-3-one ring. The packing of the molecules in the crystal structure is mainly governed by N—H···N hydrogen bonds.

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 (Raj *et al.*, 2003; Mishriky *et al.*, 1997). In this paper, the structure of the title compound, (I), is reported.



(I)

Compound (I) was synthesized by the intermolecular [3+2] cycloaddition of an azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 5-(4-methoxy)phenyl-10-(4-methoxy)benzylidene-2-(4-methoxy)-benzylidene-2,3,6,7,8,9-hexahydro-5*H*,10*H*-cyclohepta[1,2-*d*]-thiazolo[3,2-*a*]pyrimidin-3-one.

The molecular structure of (I) is illustrated in Fig. 1. In the molecule, there is a dispiro ring system, which consists of a 2-oxindole ring, a pyrrolidine ring and a 10-(arylmethylene)-hexahydrocyclohepta[1,2-*d*]thiazolo[3,2-*a*]pyrimidin-3-one ring. The pyrrolidine ring is not planar, having an envelope conformation.

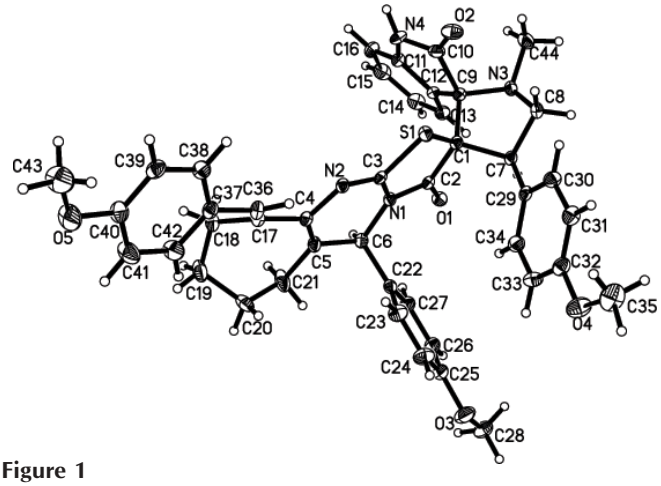


Figure 1
The molecular structure of (I), drawn with 20% probability ellipsoids. Only the major disorder component is shown.

The bond lengths and angles in (I) agree with those reported for the similar structure 10′′-(4-chlorobenzylidene)-5′′-(4-chlorophenyl)-4′-(2,4-dichlorophenyl)-1′-methyl-2,3,2′′,3′′,7′′,8′′,9′′,10′′-octahydro-1*H*,5′′*H*,6′′*H*-indole-3-spiro-2′-pyrrolidine-3′-spiro-2′′-cyclohepteno[1,2-*d*]thiazolo[3,2-*a*]-pyrimidine-2,3′′-dione (Hu *et al.*, 2005).

Pairs of molecules are connected by N—H···N hydrogen bonds (Fig. 2, Table 1).

Experimental

A mixture of 5-(4-methoxyl)phenyl-10-(4-methoxyl)benzylidene-2-(4-methoxyl)benzylidene-2,3,6,7,8,9-hexahydro-5*H*,10*H*-cyclohepta[1,2-*d*]thiazolo[3,2-*a*]pyrimidin-3-one (1 mmol), isatin (1.2 mmol) and sarcosine (1.2 mmol) was refluxed in acetonitrile (80 ml) until the disappearance of the starting materials, as evidenced by thin-layer chromatography. After completion of the reaction, the solvent was removed *in vacuo* and the residue separated by column chromatography [silica gel, petroleum ether–ethyl acetate 5:1 (*v*/*v*)] to give the title compound, (I) (m.p. 475 K). The compound was recrystallized by the dissolution of 20 mg of (I) in acetone (15 ml); the solution was kept at room temperature for 6 d allowing natural evaporation, to give colourless single crystals of (I) suitable for X-ray analysis. Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3168 (–NH), 1727, 1720 (C═O), 1378 (–CH_3); ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 1.48–2.51 (*m*, 8H, cycloheptyl), 2.20 (*s*, 3H, N—CH_3), 3.42 (*dd*, J = 7.5 and 8.7 Hz, 1H, –CH), 3.78 (*s*, 3H, O—CH_3), 3.80 (*s*, 3H, O—CH_3), 3.86 (*s*, 3H, O—CH_3), 3.99 (*dd*, J = 8.7 and 9.9 Hz, 1H, –CH), 4.10 (*dd*, J = 7.5 and 9.9 Hz, 1H, –CH), 5.15 (*s*, 1H, –CH), 6.73–7.54 (*m*, 17H, Ar-H and –CH), 8.16 (*bs*, 1H, –NH).

Crystal data

$C_{44}H_{42}N_4O_5S$	Z = 2
M_r = 738.88	D_x = 1.320 Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
a = 10.6294 (19) Å	Cell parameters from 2110 reflections
b = 13.299 (2) Å	θ = 2.3–23.1°
c = 13.702 (2) Å	μ = 0.14 mm^{-1}
α = 104.778 (3)°	T = 294 (2) K
β = 95.840 (3)°	Block, colourless
γ = 91.828 (3)°	0.28 × 0.24 × 0.20 mm
V = 1859.8 (5) Å^3	

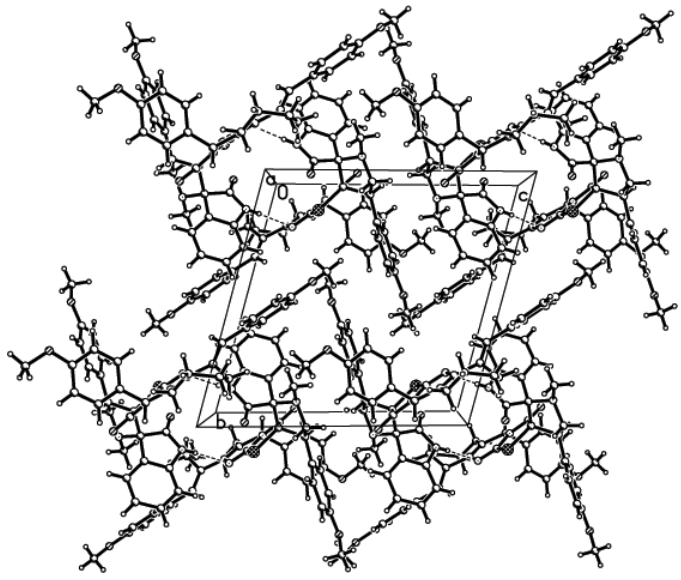


Figure 2
The crystal structure of (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

Data collection

Bruker SMART CCD area-detector diffractometer	7546 independent reflections
φ and ω scans	4011 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	R_{int} = 0.031
T_{min} = 0.958, T_{max} = 0.972	θ_{max} = 26.5°
10666 measured reflections	$h = -13 \rightarrow 11$
	$k = -16 \rightarrow 15$
	$l = -17 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 0.3384P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.055	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.140	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
S = 1.01	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
7546 reflections	
505 parameters	
H atoms treated by a mixture of independent and constrained refinement	

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···N2^{i}	0.86 (1)	2.16 (1)	3.014 (3)	173 (3)

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

Atom C20 of the cycloheptene ring is orientationally disordered over two sites (C20 and C20′), the ratio of the occupancies being 0.526 (8):0.474 (8). The H atom bound to atom N4 was refined freely. All other H atoms were positioned geometrically, with C—H = 0.93–0.98 Å, and refined in a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

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*.

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