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1′-Methyl-4′-(2,4-dichlorophenyl)-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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The title compound, $C_{21}H_{15}Cl_2N_5O_2S$, was synthesized by the intermolecular [3 + 2]-cycloaddition of the azomethine ylide derived from isatin and sarcosine by a decarboxylative route and 5-(2,4-dichlorobenzylidene)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 ring. The 2,4-dichlorophenyl group is disordered. Molecules are connected into chains by intermolecular N—H···N hydrogen bonds and weak C—H···O interactions.

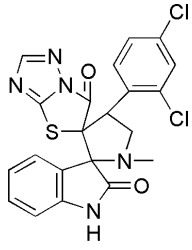
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.047
wR factor = 0.119
Data-to-parameter ratio = 13.0

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

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 an important process for the construction of spiro-compounds (Caramella & Grunanger, 1984). Here the structure of 1-methyl-spiro[2.3′]oxindole-spiro[3.″]thiazolo[3″,2″-*b*][1″,2″,4″]-azol-6″-one-4-(2,4-dichloro)-phenyl-pyrrolidine, (I), is reported.



(I)

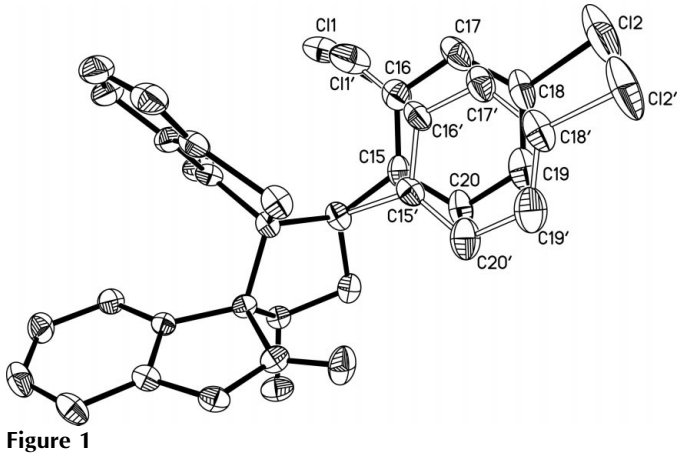


Figure 1
The molecular structure of (I), drawn with 30% probability ellipsoids. H atoms have been omitted. The minor disordered component is indicated by open bonds.

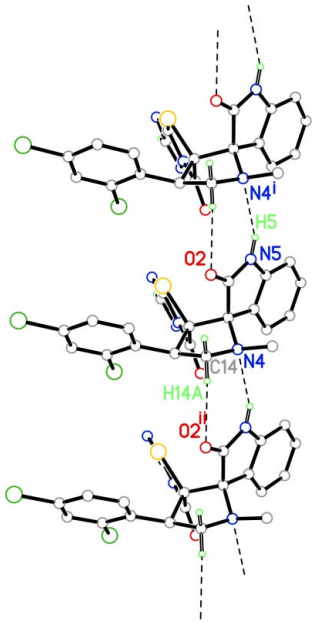


Figure 2
Part of the crystal structure of (I), showing molecules connected through unit-cell translations in the **b** direction by N—H···N hydrogen bonding and weak C—H···O interactions, which are indicated by dashed lines [symmetry codes: (i) x, $1 + y$, z; (ii) x, $y - 1$, z].

Compound (I) was synthesized by the intermolecular [3 + 2]-cycloaddition of the azomethine ylide derived from isatin and sarcosine by a decarboxylative route and 5-(2,4-dichlorobenzylidene)thiazolo[3,2-*b*][1,2,4]triazol-6-one. The molecular structure of (I) is illustrated in Fig. 1. There are two spiro junctions in the molecule, linking 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. The atoms of the 2,4-dichlorophenyl group are disordered over two sites (see Fig. 1), the ratio of occupancies being 0.542:0.458.

Molecules are connected into chains, through unit-cell translations in the *b* axis direction, by intermolecular N—H···N hydrogen bonds [N5···N5^{i} = 3.117 (3) Å, H5···N5^{i} = 2.30 Å and N5—H5···N5^{i} = 158°; symmetry code: (i) x, $1 + y$, z] and weak C—H···O interactions [C14···O2^{ii} = 3.355 Å, H14*A*···O2^{ii} = 2.52 Å and C14—H14*A*···O2^{ii} = 144°; symmetry code: (ii) x, $y - 1$, z] (see Fig. 2).

Experimental

A mixture of 5-(2,4-dichlorobenzylidene)thiazolo[3,2-*b*][1,2,4]triazol-6-one (1 mmol), isatin (1 mmol) and sarcosine (1 mmol) were refluxed in methanol (60 ml) until the starting material had disappeared, 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 = 3:1), giving the title compound, (I) (m.p. 495–496 K). IR (KBr): 3215.6 (NH), 1765.0, 1711.0 (C═O) cm^{-1}; ^{1}H NMR (p.p.m.): 2.29 (*s*, 3H, N—CH$_3$), 3.64 (*m*, 1H, –CH$_2$), 4.14 (*m*, 1H, —CH$_2$), 4.62 (*m*, 1H, —CH), 6.77–7.77 (*m*, 8H, Ar—H), 7.85 (*bs*, 1H, —NH). 20 mg of (I) was 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

$C_{21}H_{15}Cl_2N_5O_2S$	D_x = 1.485 Mg m^{-3}
M_r = 472.34	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 886 reflections
a = 20.130 (6) Å	θ = 3.2–26.0°
b = 6.357 (2) Å	μ = 0.44 mm^{-1}
c = 17.394 (5) Å	T = 293 (2) K
β = 108.369 (5)°	Block, colorless
V = 2112.6 (11) Å^3	0.24 × 0.20 × 0.10 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	4299 independent reflections
φ and ω scans	2878 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	R_{int} = 0.035
T_{min} = 0.825, T_{max} = 0.960	θ_{max} = 26.4°
11420 measured reflections	$h = -25 \rightarrow 13$
	$k = -7 \rightarrow 7$
	$l = -21 \rightarrow 21$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.047	$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.119	$(\Delta/\sigma)_{max} < 0.001$
S = 1.03	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
4299 reflections	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
330 parameters	

H atoms were positioned geometrically (C—H = 0.93–0.98 Å) and refined in the riding-model approximation [U_{iso}(H) = 1.2U_{eq} of the carrier atom or 1.5U_{eq} for methyl H atoms]. The disordered benzene ring was constrained to have the geometry of a regular hexagon, with C—C bond lengths of 1.39 (1) Å.

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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