

# 5'-(2-Chlorophenyl)-1'-methyl-2'',3''-dihydroindoline-3-spiro-3'-pyrrolidine-4'-spiro-2''-(1,3-benzimidazo[2,1-*b*]-thiazole)-2,3''-dione dioxane hemisolvate

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## Key indicators

Single-crystal X-ray study  
 $T = 293\text{ K}$   
 Mean  $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$   
 $R$  factor = 0.057  
 $wR$  factor = 0.104  
 Data-to-parameter ratio = 15.3

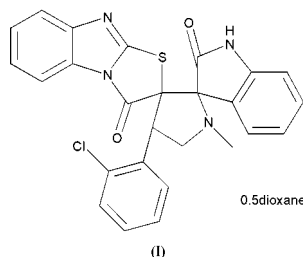
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound,  $\text{C}_{26}\text{H}_{19}\text{ClN}_4\text{O}_2\text{S}\cdot 0.5\text{C}_4\text{H}_8\text{O}_2$ , was synthesized by the intermolecular [3 + 2]-cycloaddition of azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 2-(2-chlorobenzylidene)benzo[4,5]imidazo[2,1-*b*]thiazol-3-one. In the molecule, the two spiro junctions link a planar 2-oxoindoline ring, a pyrrolidine ring in an envelope conformation, and a planar benzo[4,5]-imidazo[2,1-*b*]thiazol-3(2*H*)-one ring. Two molecules are connected by  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds to a molecule of dioxane on an inversion center, with an  $\text{N}\cdots\text{O}$  distance of 2.823 (2)  $\text{\AA}$ .

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## Comment

Spiro compounds represent an important class of naturally occurring substances characterized by pronounced biological properties (Kobayashi *et al.*, 1991; James *et al.*, 1991). 1,3-Dipolar cycloaddition reactions are important process for the construction of spiro compounds (Caramella & Grunanger, 1984). In this paper, the structure of the title compound, (I) (Fig. 1), is reported.



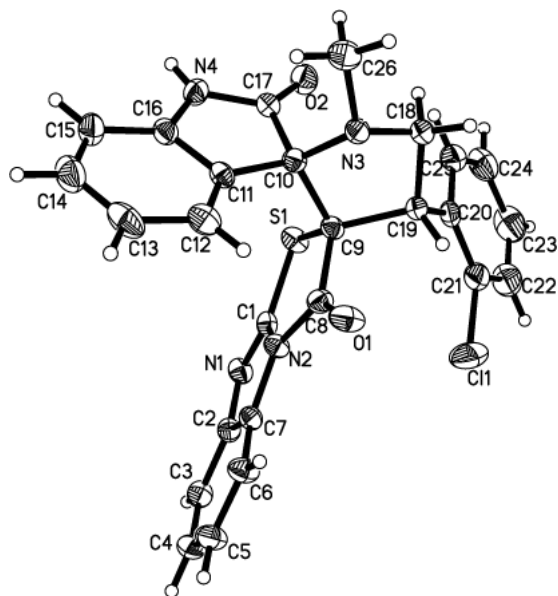
Two spiro junctions exist in the molecule, which consists of a 2-oxoindoline ring, a pyrrolidine ring and a benzo[4,5]-imidazo[2,1-*b*]thiazol-3(2*H*)-one system. The pyrrolidine ring (N3/C9/C10/C18/C19) is not planar, having an envelope conformation. Half of a dioxane solvent molecule is found in the asymmetric unit.

Two molecules of (I) are connected by  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds to the centrosymmetric dioxane molecule, with an  $\text{N}\cdots\text{O}$  distance of 2.823 (2)  $\text{\AA}$  and an  $\text{N}-\text{H}\cdots\text{O}$  angle of 150(3)°.

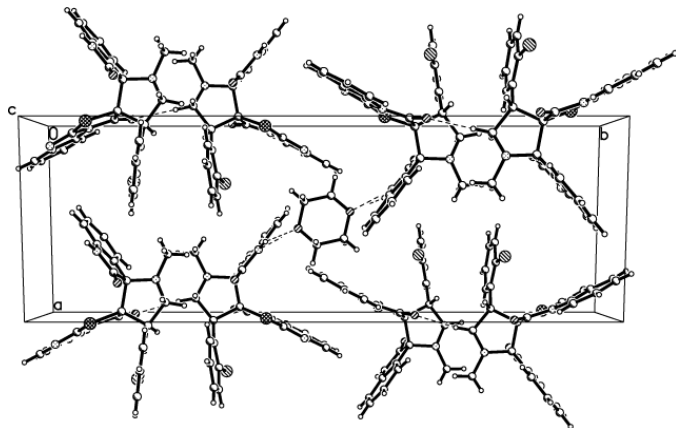
The structure of 1'-methyl-5'-phenyl-2'',3'',5'',6''-tetrahydroindoline-3-spiro-3'-pyrrolidine-4'-spiro-2''-imidazo[2,1-*b*]thiazole-2,3''-dione has been reported previously (Li *et al.*, 2003).

## Experimental

A mixture of 2-(2-chlorobenzylidene)benzo[4,5]imidazo[2,1-*b*]thiazol-3-one, isatin (1 mmol) and sarcosine (1 mmol) was refluxed in methanol (60 ml) until total consumption of the starting material, as



**Figure 1**  
The molecular structure of (I), showing the atom-numbering scheme, drawn with 30% probability ellipsoids.



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

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 the title compound (I). M.p.: 516–518 K; IR (KBr): 1751.5, 1685.9 (C=O), 1612.6 (C=N)  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ,  $\delta$ , p.p.m.): 2.32 (3H, s), 3.67 (1H, m), 4.24 (1H, m), 4.69 (1H, m), 6.69–7.87 (12H, m), 7.89 (1H, br). 20 mg of (I)

was dissolved in 15 ml dioxane and the solution was kept at room temperature for 10 d. Natural evaporation afforded colorless single crystals of (I), suitable for X-ray analysis.

#### Crystal data

$\text{C}_{26}\text{H}_{19}\text{ClN}_4\text{O}_2\text{S}\cdot 0.5\text{C}_4\text{H}_8\text{O}_2$   
 $M_r = 531.01$   
 Monoclinic,  $P2_1/c$   
 $a = 10.085$  (3) Å  
 $b = 29.272$  (9) Å  
 $c = 8.561$  (3) Å  
 $\beta = 99.287$  (6)°  
 $V = 2494.2$  (13) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.414$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 911 reflections  
 $\theta = 2.5$ – $24.6^\circ$   
 $\mu = 0.28$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Block, colorless  
 $0.18 \times 0.16 \times 0.14$  mm

#### Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Bruker, 1997)  
 $T_{\min} = 0.924$ ,  $T_{\max} = 0.960$   
 14310 measured reflections

5109 independent reflections  
 2883 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.069$   
 $\theta_{\max} = 26.4^\circ$   
 $h = -11 \rightarrow 12$   
 $k = -36 \rightarrow 34$   
 $l = -8 \rightarrow 10$

#### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.057$   
 $wR(F^2) = 0.104$   
 $S = 1.02$   
 5109 reflections  
 335 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.21$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.25$  e Å<sup>-3</sup>

H atoms were placed at geometrically calculated positions (C–H = 0.93–0.98 Å and N–H = 0.86 Å) and were included in the refinement in riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier atom})$  or  $1.5U_{\text{eq}}$  for methyl atoms.

Data collection: SMART (Bruker, 1997); cell refinement: SMART; data reduction: SAINT (Bruker, 1997); 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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