Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4′-(2,4-Dichlorophenyl)-1′-methyl-2,3,2″,3″-tetrahydro-1*H*-indole-3-spiro-2′-pyrrolidine-3′-spiro-2″-(1,3-benzimidazo[2,1-*b*]thiazole)-2,3″-dione

Xiao-Fang Li,* Ya-Qing Feng, Da-Xin Shi and Hong-Liang Chen

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

Correspondence e-mail: lxf7212@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.050
wR factor = 0.107
Data-to-parameter ratio = 15.4

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

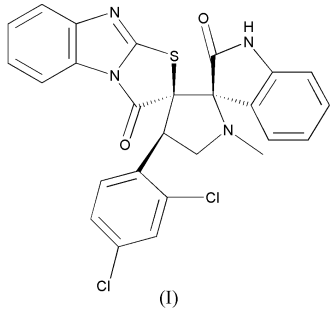
© 2003 International Union of Crystallography
Printed in Great Britain – all rights reserved

Received 15 August 2003
Accepted 21 August 2003
Online 30 August 2003

In the title compound, $C_{26}H_{18}Cl_2N_4O_2S$, the two spiro junctions link a planar 2-oxindole ring, a pyrrolidine ring in an envelope conformation and a planar benzo[4,5]-imidazo[2,1-*b*]thiazol-3-one ring. Molecules form dimers connected by N—H···N hydrogen bonds.

Comment

Spiro compounds represent an important class of naturally occurring substances characterized by highly pronounced biological properties (James *et al.*, 1991; Kobayashi *et al.*, 1991). 1,3-Dipolar cycloaddition reactions are important processes for the construction of spiro compounds (Caramella & Grunanger, 1984). We reported previously the structure of 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 (Li *et al.*, 2003). In the present paper, the structure of a related compound, *viz*. the title compound, (I), is reported. Compound (I) was synthesized by the intermolecular [3+2]-cycloaddition of azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 2-(2,4-dichlorobenzylidene)benzo[4,5]imidazo[2,1-*b*]thiazol-3-one.



(I)

The molecular structure of (I) is illustrated in Fig. 1. The molecule contains dispiro rings that consist of a 2-oxindole ring, a pyrrolidine ring and a benzo[4,5]imidazo[2,1-*b*]thiazol-3-one ring. The pyrrolidine ring (N3/C11/C12/C1/C10) exhibits an envelope conformation, with atom C10 in the flap position. Two molecules related by a center of symmetry are connected by N—H···N hydrogen bonds (Table 1). There also exists a short intermolecular Cl2···N1 contact [3.258 (4) Å; Fig. 2].

Experimental

A mixture of 2-(2,4-dichlorobenzylidene)benzo[4,5]imidazo[2,1-*b*]-thiazol-3-one (1 mmol), isatin (1 mmol) and sarcosine (1 mmol) was refluxed in methanol (60 ml) until the disappearance of the starting material was evidenced by thin-layer chromatography. After

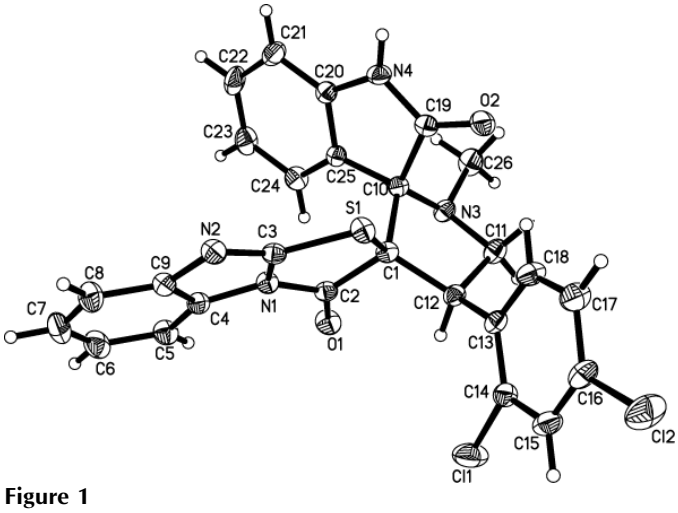


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level and the atom-numbering scheme.

evaporation of the solvent, the residue was separated by column chromatography (silica gel; petroleum ether/ethyl acetate 5:1) to give (I) (m.p. 502–503 K). IR (KBr, cm^{-1}): 1755.5, 1684.2 (C═O), 1612.8 (C═N); ^{1}H-NMR ($CDCl_3$, p.p.m.): 2.31 (3H, *s*), 3.68 (1H, *m*), 4.26 (1H, *m*), 4.73 (1H, *m*), 6.78–7.71 (11H, *m*), 7.83 (1H, *br*). Compound (I) (20 mg) was dissolved in dioxane (15 ml) and the solution was left to evaporate at room temperature for 10 d, giving colorless single crystals of (I) suitable for X-ray analysis.

Crystal data

$C_{26}H_{18}Cl_2N_4O_2S$
M_r = 521.40
Monoclinic, $P2_1/n$
a = 13.209 (6) Å
b = 11.107 (5) Å
c = 17.248 (8) Å
β = 109.673 (7)°
V = 2382.8 (19) Å^3
Z = 4
D_x = 1.453 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 856 reflections
θ = 2.4–26.4°
μ = 0.39 mm^{-1}
T = 293 (2) K
Plate, colorless
0.24 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.908, T_{max} = 0.932
13 265 measured reflections
4889 independent reflections
3385 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 26.5°
$h = -16 \rightarrow 16$
$k = -9 \rightarrow 13$
$l = -20 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.107
S = 1.09
4889 reflections
317 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

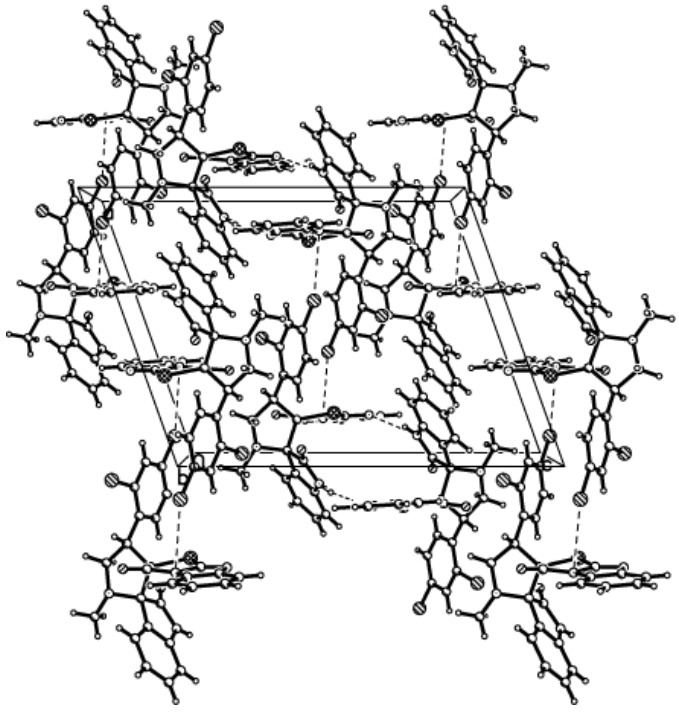


Figure 2
The crystal packing structure of (I), viewed along the *b* axis. The dashed lines represent N—H···N hydrogen bonds and Cl···N intermolecular short contacts.

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···N2^{i}	0.86	2.08	2.918 (3)	164

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

H atoms were positioned geometrically and allowed for using a riding model [C—H = 0.93–0.98 Å and N—H = 0.86 Å, and U_{iso}(H) = 1.5U_{eq}(C) (methyl H atoms) and 1.2U_{eq}(parent atom) (other H atoms)].

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*, edited by A. Padwa, Vol. 1, 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. & Mikami, Y. (1991). *Tetrahedron*, **47**, 6617–6622.
Li, X.-F., Feng, Y.-Q., Li, G. & Wang, G. (2003). *Acta Cryst.* E**59**, o1307–o1308.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.