Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

1′-Methyl-5′-phenyl-2″,3″,5″,6″-tetrahydroindoline-3-spiro-3′-pyrrolidine-4′-spiro-2″-imidazo[2,1-*b*]thiazole-2,3″-dione

Xiao-Fang Li,* Ya-Qing Feng, Da-Xin Shi and Gang Li

School of Chemical Engineering and Technology, Tianjin University, The State Key Laboratory of C1 Chemical Technology, Tianjin University, Tianjin 300072, People's Republic of China

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

Received 25 July 2003
Accepted 6 August 2003
Online 8 August 2003

The title compound, $C_{22}H_{20}N_4O_2S$, was synthesized by the intermolecular [3 + 2]-cycloaddition of azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 2-benzylidene-5,6-dihydro-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 5,6-dihydroimidazo[2,1-*b*]thiazol-3(2*H*)-one ring. Two molecules are connected into a dimer by two N—H···N hydrogen bonds.

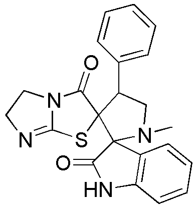
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.101
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.

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 (Caramella & Grunanger, 1984). In this paper, the structure of the title compound, (I) (Fig. 1), is reported.



(I)

Two spiro junctions exist in the molecule, which consists of a 2-oxoindoline ring, a pyrrolidine ring and a 5,6-dihydroimidazo[2,1-*b*]thiazol-3(2*H*)-one ring. The pyrrolidine ring (N3/C6/C5/C15/C14) is not planar, with an envelope conformation. Atoms C6/C5/C15/C14 are almost coplanar, the mean deviation from this plane being 0.038 (3) Å. Atom N3 lies 0.609 (3) Å above the C6/C5/C15/C14 plane in the pyrrolidine ring, forming the flap of the envelope. The dihedral angle between the C6/N3/C14 plane and the C6/C5/C15/C14 mean plane is 44.4 (2)°. The dihedral angle between the phenyl plane (C16–C21) and the C6/C5/C15/C14 plane is 97.8 (2)°. The 2-oxoindoline ring (C6–C13/N4) is nearly planar, the mean deviation from this plane being 0.032 (3) Å. The dihedral angle between the 2-oxoindoline ring mean plane and the C6/C5/C15/C14 plane is 101.0 (2)°. The dihedral angle between the 5,6-dihydro-imidazo[2,1-*b*]thiazol-3(2*H*)-one plane and the C6/C5/C15/C14 plane is 91.7 (3)°.

Two molecules are connected into a dimer by two N—H···N hydrogen bonds, with an N···N distance of 2.904 (2) Å and an N—H···N angle of 169°.

DOI: 10.1107/S160053680301746X

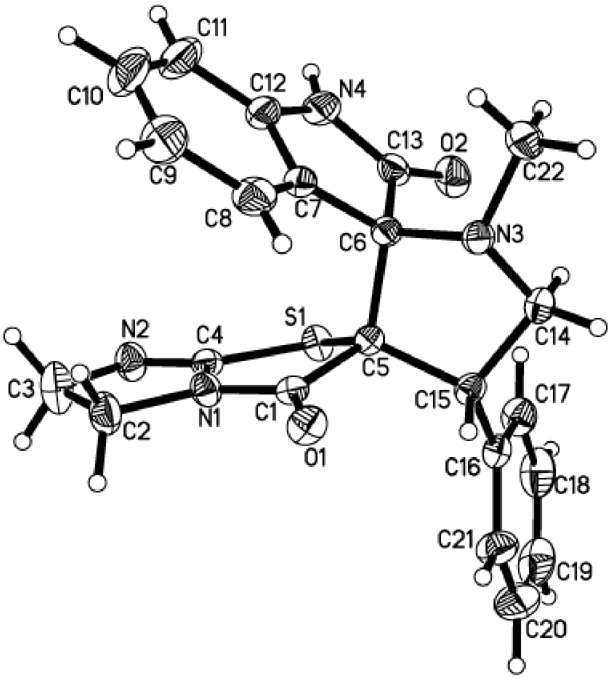


Figure 1
The molecular structure of (I), showing the atom-numbering scheme, drawn with 30% probability ellipsoids.

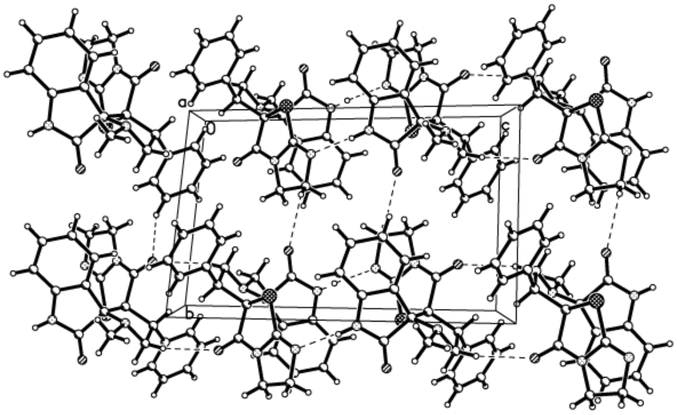


Figure 2
The crystal packing diagram of (I), viewed along the *a* axis

Experimental

A mixture of 2-benzylidene-5,6-dihydroimidazo[2,1-*b*]thiazol-3-one (1 mmol), isatin (1 mmol) and sarcosine (1 m mol) was refluxed in methanol (60 ml) until the starting material had disappeared, as evidenced by thin-layer chromatography. After the reaction was over the solvent was removed *in vacuo* and the residue was separated by column chromatography (silica gel, petroleum ether/ethylacetate = 2:1) to give the title compound, (I). M.p.519–521 K; IR (KBr): 3352.3 (—NH), 1721.3, 1686.1 (C═O) cm^{-1}; ^{1}H NMR (δ, p.p.m.): 1.67 (*s*, 4H, CH$_2$), 2.27 (*s*, 3H, N-CH$_3$), 3.29 (*m*, 1H, —CH$_2$), 3.92 (*m*, 1H, —CH$_2$), 4.61 (*m*, 1H, —CH), 6.81–7.52 (*m*, 9H, ArH), 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 afforded colorless single crystals of (I) suitable for X-ray analysis.

Crystal data

$C_{22}H_{20}N_4O_2S$
$M_r = 404.48$
Triclinic, $P\overline{1}$
$a = 8.538$ (3) Å
$b = 9.488$ (3) Å
$c = 14.227$ (5) Å
$\alpha = 86.206$ (6)°
$\beta = 73.315$ (6)°
$\gamma = 64.091$ (5)°
$V = 990.7$ (6) Å^3

$Z = 2$
$D_x = 1.356$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 875 reflections
$\theta = 2.4$–26.3°
$\mu = 0.19$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.26 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
$T_{\min} = 0.938$, $T_{\max} = 0.960$
5754 measured reflections

4015 independent reflections
3129 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.015$
$\theta_{\max} = 26.4$°
$h = -9 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.101$
$S = 1.03$
4015 reflections
263 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}

All H atoms were placed in calculated positions, with C—H distances ranging from 0.93 to 0.98 Å and and an N—H distance of 0.86 Å. These atoms were included in the refinement in riding-motion approximation, with $U_{\rm iso}$ = 1.2 (1.5 for methyl) times $U_{\rm eq}$ of the carrier atom.

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*, Vol. 1, edited by A. Padwa, pp. 291–312. New York: Wiley.

James, D. M., Kunze, H. B. & Faulkner, D. J. (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.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.