

1-Methyl-spiro[2.3']oxindole-spiro[3.2']-5'',6''-dihydroimidazo[2'',1''-b]thiazol-3''-one-4-(2-benzo[1,3]dioxol-5-yl)pyrrolidine

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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

R factor = 0.034

wR factor = 0.107

Data-to-parameter ratio = 15.1

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

The title compound, $\text{C}_{23}\text{H}_{20}\text{N}_4\text{O}_4\text{S}$, was synthesized by the intermolecular [3 + 2] cycloaddition of azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 2-benzo[1,3]dioxol-5-ylmethylene-5,6-dihydro-imidazo[2,1-*b*]thiazol-3-one. In the molecule, two spiro junctions link a planar 2-oxindole ring, a pyrrolidine ring in an envelope conformation and a 5,6-dihydro-imidazo[2,1-*b*]thiazol-3-one ring. Two molecules are connected into a dimer by two N—H···N hydrogen bonds.

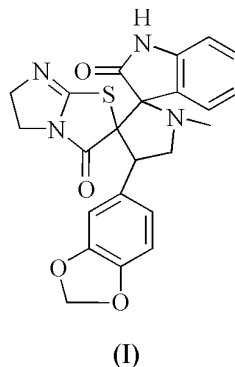
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Comment

Spiro-compounds are 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, 1-methyl-spiro[2.3']oxindole-spiro[3.2']5'',6''-dihydroimidazo[2'',1''-*b*]thiazol-3''-one-4-(2-benzo[1,3]dioxol-5-yl)pyrrolidine, (**1**), is reported. The title compound was synthesized by the intermolecular [3 + 2] cycloaddition of azomethine ylide, derived from isatin and sarcosine by a decarboxylative route, and 2-benzo[1,3]dioxol-5-ylmethylene-5,6-dihydro-imidazo[2,1-*b*]thiazol-3-one. The molecular structure of (**1**) is illustrated in Fig. 1. There are two spiro junctions in the molecule, which consists of a 2-oxindole ring, a pyrrolidine ring and a benzo[4,5]imidazo[2,1-*b*]thiazol-3-one ring. The pyrrolidine ring is not planar, having an envelope conformation. Two molecules are connected by N—H···N hydrogen bonds (Fig. 2), with an N···N distance of 2.939 (2) Å and an N—H···N angle of 175°. The structure of 1-methyl-spiro[2.3']oxindole-spiro[3.2']5'',6''-dihydroimidazo[2'',1''-*b*]thiazol-3''-one-4-phenylpyrrolidine was reported previously (Li *et al.*, 2003).



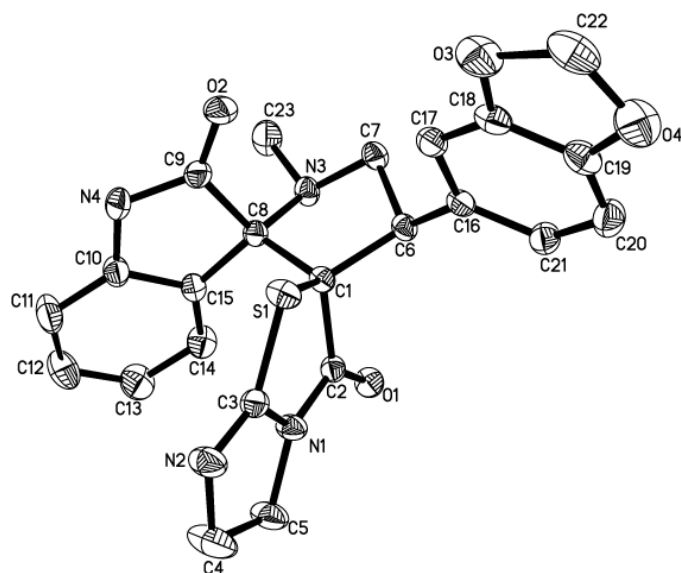


Figure 1
The molecular structure of (I), with 30% probability ellipsoids. H atoms have been omitted for clarity.

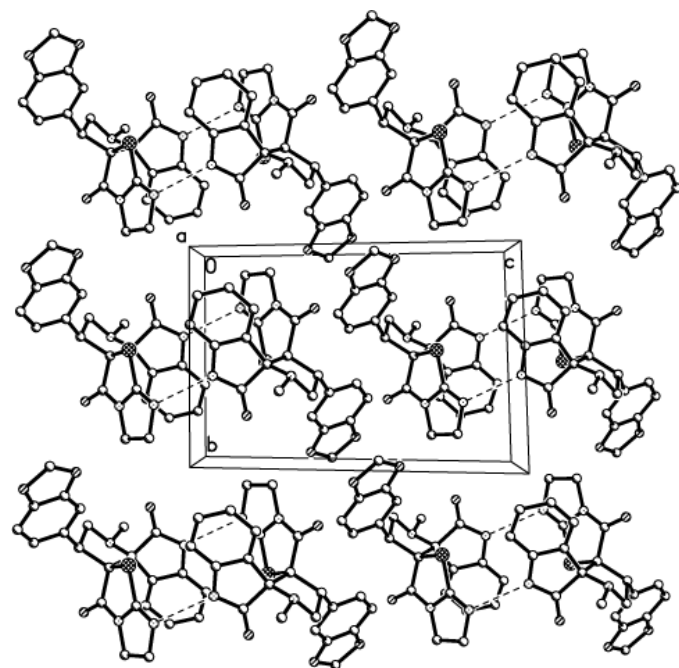


Figure 2
The crystal structure of (I), viewed along the *a* axis. Hydrogen bonds are indicated by dashed lines. H atoms are omitted for clarity.

Experimental

A mixture of 2-benzo[1,3]dioxol-5-ylmethylene-5,6-dihydroimidazo[2,1-*b*]thiazol-3-one (1 mmol), isatin (1 mmol) and sarcosine (1 mmol) was refluxed in methanol (60 ml) until the starting materials had disappeared, as evidenced by TLC. After the reaction

was over, the solvent was removed *in vacuo* and the residue was separated by column chromatography (silica gel, petroleum ether/ethyl acetate 2:1) to give the title compound (I). m.p. 543–544 K; IR (KBr): 3352.0 (–NH), 1720.4, 1686.7 (C=O) cm^{-1} ; $^1\text{H-NMR}$ (δ , p.p.m.): 1.69 (*s*, 4H, CH_2), 2.26 (*s*, 3H, N– CH_3), 3.29 (*m*, 1H, – CH_2), 4.33 (*m*, 1H, – CH_2), 5.42 (*m*, 1H, –CH), 5.91 (*s*, 2H, – CH_2), 6.41–7.73 (*m*, 7H, Ar–H), 7.89 (*bs*, 1H, –NH). 20 mg of (I) were dissolved in 15 ml dioxane and the solution kept at room temperature for 15 d. Natural evaporation afforded colorless single crystals of (I), suitable for X-ray analysis.

Crystal data

$\text{C}_{23}\text{H}_{20}\text{N}_4\text{O}_4\text{S}$
 $M_r = 448.49$
Triclinic, $P\bar{1}$
 $a = 8.741$ (4) Å
 $b = 9.255$ (4) Å
 $c = 14.097$ (7) Å
 $\alpha = 85.690$ (7)°
 $\beta = 72.206$ (7)°
 $\gamma = 79.169$ (8)°
 $V = 1066.4$ (9) Å³

$Z = 2$
 $D_x = 1.397$ Mg m^{–3}
Mo $K\alpha$ radiation
Cell parameters from 1010 reflections
 $\theta = 2.5$ – 26.5 °
 $\mu = 0.19$ mm^{–1}
 $T = 293$ (2) K
Block, colorless
0.28 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector
diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 1997)
 $T_{\min} = 0.863$, $T_{\max} = 0.960$
14787 measured reflections

4388 independent reflections
3798 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.019$
 $\theta_{\max} = 26.5$ °
 $h = -10 \rightarrow 10$
 $k = -11 \rightarrow 11$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.107$
 $S = 1.09$
4388 reflections
290 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.29$ e Å^{–3}
 $\Delta\rho_{\min} = -0.21$ e Å^{–3}

H atoms were positioned geometrically (C–H = 0.93–0.98 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$.

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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