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7-Phenylcyclooctane-1-spiro-6′-2-thiapyrrolizidine-5′-spiro-3″-indole-2,2″(3*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.047
wR factor = 0.136
Data-to-parameter ratio = 17.4

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

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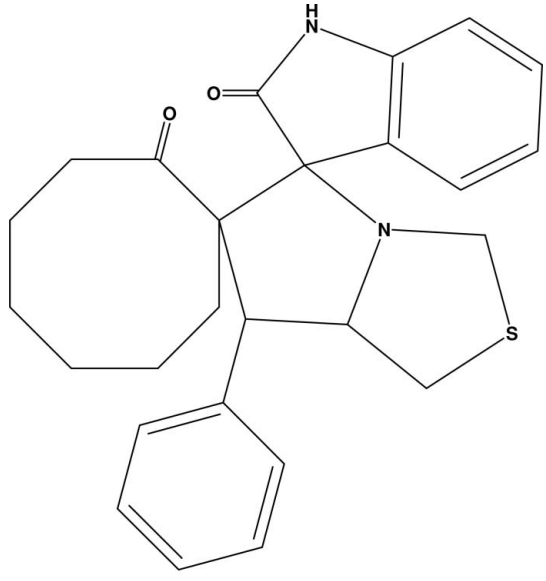
In the title compound, $C_{26}H_{28}N_2O_2S$, the pyrrolidine ring adopts a twisted conformation and the thiazolidine ring is in an envelope conformation. The eight-membered ring adopts a twist-boat-chair conformation. The molecular structure is stabilized by a weak C—H···O interaction and the crystal packing is stabilized by intermolecular N—H···O interactions, generating a centrosymmetric dimer with an $R_2^2(8)$ motif.

Comment

Pyrrolidine has gained much attention in the pharmacological industry for its high medicinal value. Pyrrolidine compounds have antifungal and antimicrobial activity (Amal Raj *et al.*, 2003). The spiro ring system is frequently encountered in many pharmacologically relevant alkaloids. Synthetic spiro-pyrrolidine derivatives have activity against the aldose reductase enzyme which controls influenza (Stylianakis *et al.*, 2003). Owing to the high medicinal importance of spiro-pyrrolidine derivatives, we have undertaken the X-ray crystal structure determination of the title compound, (I).



(I)

Fig. 1 shows the molecular structure of compound (I). Bond lengths and angles are comparable with reported values (Allen *et al.*, 1987), except for the bonds at the spiro junctions (Table 1). The sum of the bond angles around atom N1 (341.1°) indicates sp^3 hybridization. The five-membered ring of the oxindole system is planar and atom O1 deviates from this plane by 0.101 (2) Å. The dihedral angle between the five- and six-membered rings in the oxindole system is 4.6 (1)°.

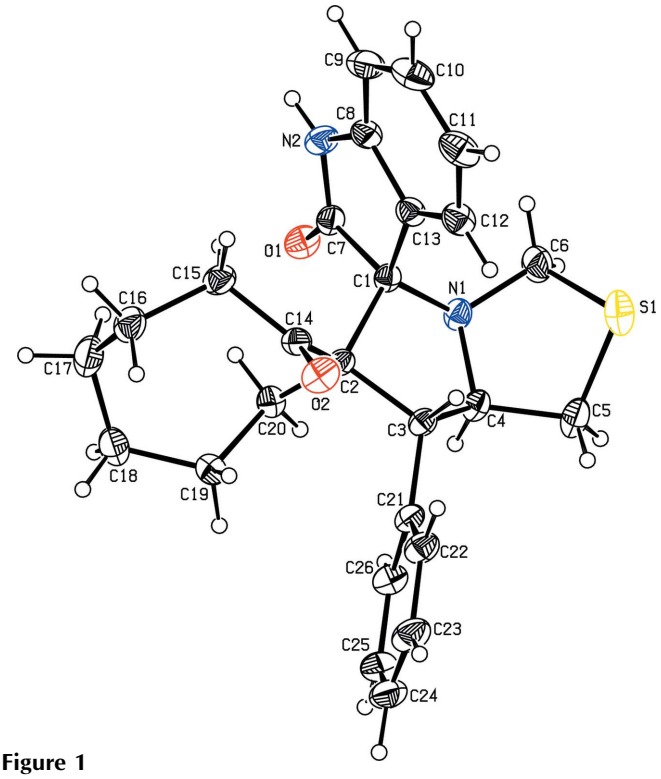


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

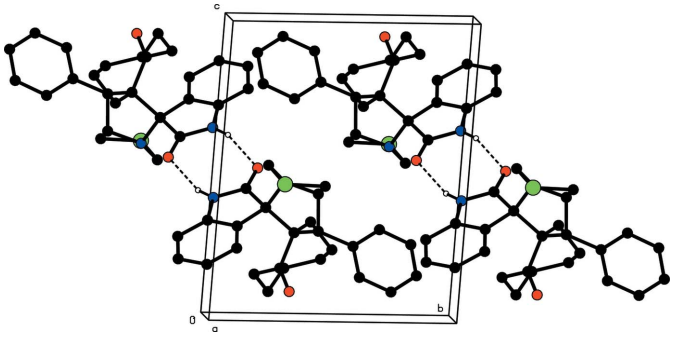


Figure 2
The packing of (I), viewed approximately down the *a* axis. For the sake of clarity, H atoms not involved in hydrogen bonds (dashed lines) have been omitted.

The pyrrolidine ring adopts a twisted conformation, with a pseudo-twofold axis passing through atom N1 and the midpoint of the C2—C3 bond. The thiazolidine ring adopts an envelope conformation, with atom S1 deviating 0.802 (1) Å from the least-squares plane passing through the remaining four atoms of that ring. The puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) for the pyrrolidine ring are q_2 = 0.424 (1) Å, φ = 82.9 (2)° and $\Delta_2(N_1)$ = 8.0 (2), respectively, and those for the thiazolidine ring are q_2 = 0.466 (2) Å, φ = 358.8 (2)° and $\Delta_s(S_1)$ = 2.6 (1), respectively. The eight-membered ring adopts a twist–boat–chair conformation.

A weak C—H···O interaction is observed in the molecular structure. The crystal packing is stabilized by N2—H2···O1($-x, -y, 1-z$) intermolecular interactions (Table 2), generating a centrosymmetric dimer of $R_2^2(8)$ motif (Bernstein *et al.*, 1995) (Fig. 2).

Experimental

A mixture of isatin (1.2 mmol), thiazolidine-4-carboxylic acid (1.2 mmol) and benzylidene cyclooctanone (1 mmol) in methanol (20 ml) was refluxed until the disappearance of the starting materials, as shown by thin-layer chromatography. The solvent was then evaporated *in vacuo* and the residue was chromatographed on a column (silica gel, 100–200 mesh) eluted with a hexane–ethyl acetate (9:1 *v*/*v*) mixture to obtain the title compound, which was recrystallized from a solution in methanol.

Crystal data

$C_{26}H_{28}N_2O_2S$
M_r = 432.56
Triclinic, $P\overline{1}$
a = 9.0901 (6) Å
b = 10.5013 (7) Å
c = 12.7508 (8) Å
α = 79.865 (1)°
β = 71.504 (1)°
γ = 68.568 (1)°
V = 1071.99 (12) Å^3
Z = 2
D_x = 1.340 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.18 mm^{-1}
T = 293 (2) K
Block, colourless
0.26 × 0.25 × 0.22 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
12269 measured reflections
4878 independent reflections
4378 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.136
S = 1.07
4878 reflections
280 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 0.2495P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.470 (2)	C3—C4	1.529 (2)
C1—C13	1.531 (2)	C4—N1	1.469 (2)
C1—C7	1.564 (2)	C4—C5	1.531 (2)
C1—C2	1.591 (2)	C5—S1	1.801 (2)
C2—C14	1.538 (2)	C6—N1	1.445 (2)
C2—C20	1.545 (2)	C6—S1	1.835 (2)
C2—C3	1.560 (2)	C7—O1	1.217 (2)
C3—C21	1.517 (2)	C14—O2	1.208 (2)
C6—N1—C4	111.7 (1)	C7—N2—C8	112.0 (1)
C6—N1—C1	118.1 (1)	C5—S1—C6	87.6 (1)
C4—N1—C1	111.3 (1)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.86	2.06	2.879 (2)	160
C20—H20B···O1	0.97	2.42	3.017 (2)	119

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with N—H = 0.86 Å and C—H = 0.93–0.98 Å, and U_{iso}(H) = 1.2 U_{eq}(C,N).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Amal Raj, A., Raghunathan, R., Sridevi Kumari, M. R. & Raman, N. (2003). *Bioorg. Med. Chem.* **11**, 407–409.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2001). *SMART* (Version 5.625/NT/2000) and *SAINT* (Version 6.28a). Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Nardelli, M. (1983). *Acta Cryst.* C**39**, 1141–1142.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stylianakis, I., Kolocouris, A., Kolocouris, N., Fytas, G., Foscolos, G. B., Padalko, E., Neyts, J. & De Clercq, E. (2003). *Bioorg. Med. Chem. Lett.* **13**, 1699–1703.