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1′-Phenyl-2′,3′,5′,6′,7′,7a′-hexahydroindan-2-spiro-2′-1*H*-pyrrolizine-3′-spiro-11′-indeno[1,2-*b*]quinoxaline-1,3-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 = 0.128
Data-to-parameter ratio = 16.7

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

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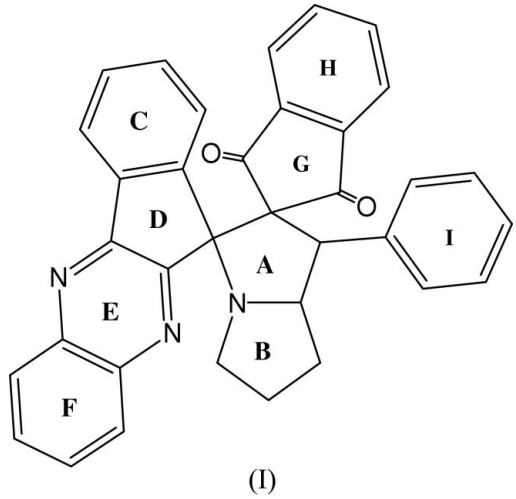
The title compound, $C_{27}H_{22}N_2O_3$, comprises an indeno-quinoxaline system linked *via* a spiro ring junction to a hexahydro-1*H*-pyrrolizine unit. This in turn carries an indene-1,3-dione linked again by a spiro junction through the five-membered indenone ring. The pyrrolizidine moiety is folded and twisted about the N—C bond common to the two five-membered rings. In the crystal packing, an $R_2^2(20)$ graph set involves a dimeric C—H···O hydrogen bond and ring motif. The packing is further stabilized by C—H···O and weak π–π interactions.

Comment

Pyrrolidine, the saturated tetrahydropyrrole, is a basic intermediate used in wide range of applications in organic synthesis. It has also gained much attention in the pharmacological industry for its medicinal value. Derivatives of pyrrolidine are found to have anticonvulsant (Obniska *et al.*, 2002), antimicrobial and antifungal activity against various pathogens, except Bacillus subtilis (Amal Raj *et al.*, 2003). Quinoxaline derivatives show antibacterial, antiviral and anticancer properties (Zarranz *et al.*, 2003). The spiro ring system is a frequently encountered structural motif in many pharmacologically relevant alkaloids. Synthetic spiro-pyrrolidine derivatives have activity against the aldose reductase enzyme which controls influenza (Stylianakis *et al.*, 2003). As spiro-pyrrolidine compounds are of great medicinal importance, we have undertaken the three-dimensional structure determination of the title compound, (I), by X-ray diffraction (Fig. 1).



(I)

The sum of the bond angles at N1 of the pyrrolidine ring (340.4°) indicates sp^3 hybridization. The C14═N2 and

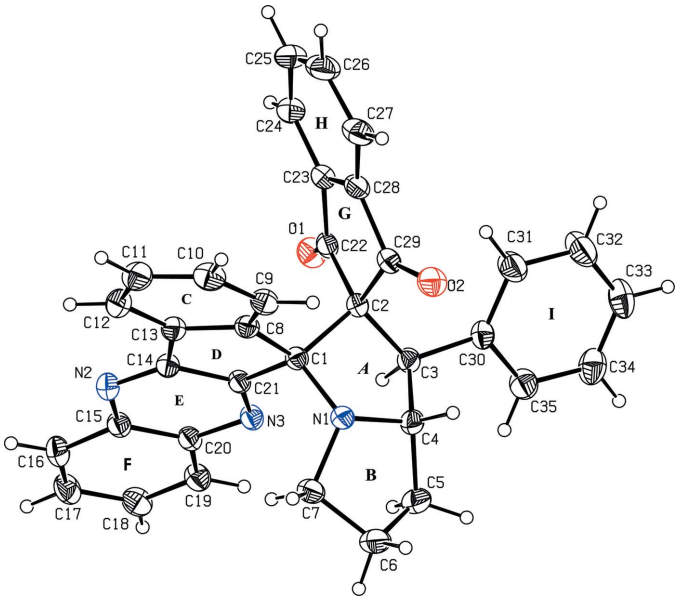


Figure 1
The molecular structure of (I), showing 35% probability displacement ellipsoids.

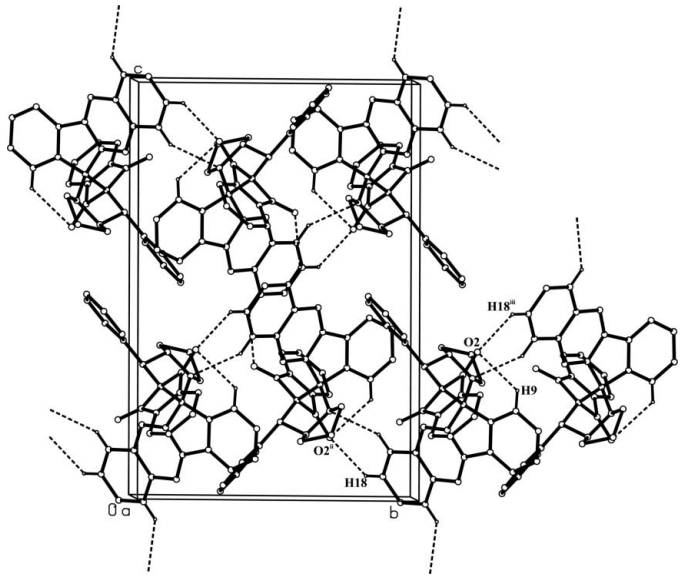


Figure 2
The molecular packing of (I), viewed down the *a* axis, with hydrogen bonds drawn as dashed lines. For the sake of clarity, H atoms not involved in the hydrogen bonds have been omitted.

C21═N3 bond distances are comparable with other reported values (Allen *et al.*, 1987). The spiro junction at C2 in the indanedione group deviates from the mean plane (C22–C29) by 0.290 (1) Å. Atoms O1 and O2 deviate from the mean plane of the indanedione ring by −0.240 (1) and −0.351 (1) Å, respectively. Atom O2 displays a greater deviation than atom O1, as it is involved in both intra- and intermolecular interactions, whereas atom O1 only forms an intermolecular hydrogen bond (Table 2).

In the pyrrolizidine system (*A*/*B*), rings *A* and *B* adopt envelope and twist conformations, respectively. The puckering parameters (Cremer & Pople, 1975) and smallest displacement asymmetry parameters (Nardelli, 1983) are, for ring *A*, N1/C1–C4, q_2 = 0.445 (1) Å, φ = 72.5 (2)° and Δ_s(C2) = 0.003 (1), and for ring *B*, N1/C4–C7, q_2 = 0.401 (2) Å, φ = −83.7 (2)°, ΔC_2(N1) = 0.026 (1). The pyrrolizidine moiety is twisted and folded about the N1—C4 bond, as indicated by the torsion angles C7—N1—C4—C5 [8.2 (1)°] and C7—N1—C4—H4 [−108.2 (1)°]; a similar conformation was reported by Usha *et al.* (2005).

In the crystal packing, atom O2 is involved in both intra- and intermolecular hydrogen bonding and acts as a bifurcated acceptor; the angle C9···O2···C18^{i} between the donors is 85.0 (4)° [symmetry code: (i) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$]. The translated molecules are linked by C18—H18···O2 and C19—H19···N1 hydrogen bonds, forming a binary graph set $R_2^2(9)$ (Bernstein *et al.*, 1995). Hence, a zigzag pattern is formed by chains parallel to the *b* axis. Weak intermolecular π–π interactions occur between the stacked pyrazine and benzene rings, with a centroid separation of 3.866 (1) Å. These, together with a dimeric C16–H16···O1 hydrogen bond, stabilize the structure further.

Experimental

Ninhydrin (1 mmol), *o*-phenylenediamine (1 mmol), 2-benzylidene–1,3-indanedione (1 mmol) and L-proline (1 mmol) were refluxed in methanol until the starting material disappeared. The crude product was purified by column chromatography (petroleum ether–ethyl acetate, 8:2) and the product, compound (I), was re-crystallized from methanol.

Crystal data

$C_{35}H_{25}N_3O_2$
M_r = 519.58
Monoclinic, $P2_1/c$
a = 11.1149 (14) Å
b = 12.4670 (16) Å
c = 18.646 (2) Å
β = 99.415 (2)°
V = 2548.9 (6) Å^3
Z = 4
D_x = 1.354 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2836 reflections
θ = 2.3–25.2°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.25 × 0.22 × 0.20 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: none
28930 measured reflections
6030 independent reflections
4851 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 28.0°
$h = -14 \rightarrow 14$
$k = -16 \rightarrow 16$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.128
S = 1.03
6030 reflections
361 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0697P)^2 + 0.4648P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.460 (2)	C20—N3	1.381 (2)
C4—N1	1.480 (2)	C21—N3	1.298 (2)
C7—N1	1.472 (2)	C22—O1	1.205 (2)
C14—N2	1.306 (2)	C29—O2	1.202 (2)
C1—N1—C7	119.5 (1)	C7—N1—C4	109.9 (1)
C1—N1—C4	111.1 (1)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···N3	0.98	2.49	3.178 (2)	127
C9—H9···O2	0.93	2.54	3.229 (2)	131
C16—H16···O1^{i}	0.93	2.51	3.252 (2)	137
C18—H18···O2^{ii}	0.93	2.57	3.344 (2)	141
C19—H19···N1^{ii}	0.93	2.46	3.275 (2)	147

Symmetry codes: (i) $-x+1, -y+2, -z$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

The crystals were weakly diffracting, particularly at high θ values. All H atoms were fixed geometrically and allowed to ride on their parent C atoms, with C—H distances fixed in the range 0.93–0.97 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

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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In the paper by Gayathri, Aravindan, Velmurugan, Ravikumar & Sureshbabu [*Acta Cryst.* (2005), E**61**, o3124–o3126], the formula is given incorrectly in the *Abstract*. The correct formula is $C_{35}H_{25}N_3O_2$.