Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

5-Benzylidene-1-phenyl-2″,3″-dihydropiperidine-3-spiro-2′-pyrrolizidine-3′-spiro-3″-1*H*-indole-4,2″-dione

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Received 27 March 2007
Accepted 28 March 2007

Key indicators

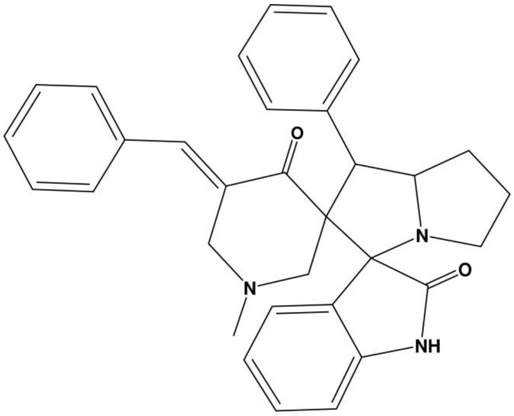
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.052
wR factor = 0.142
Data-to-parameter ratio = 18.6

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

In the pyrrolizidine unit of the title compound, $C_{32}H_{31}N_3O_2$, one pyrrolidine ring adopts an envelope conformation and the other adopts a twist conformation. The molecular conformation is stabilized by weak C—H···O intramolecular interactions and the crystal packing is stabilized by N—H···O and C—H···O hydrogen bonds.

Comment

Pyrrolidines are common structural motifs in drugs and drug candidates, showing antimicrobial and antifungal activity (Amal Raj *et al.*, 2003). Oxindole derivatives are found to be potent aldose reductase inhibitors (ARIs), which help to treat and prevent diabetic complications arising from elevated levels of sorbitol (Rajeswaran *et al.*, 1999).



(I)

The geometric parameters of the title compound are comparable with the literature values (Allen *et al.*, 1987), except at the spiro junctions. The sum of the bond angles around N1 [340.5°] and N3 [334.2°] indicates sp^3 hybridization of these N atoms. The two phenyl rings (C18–C23 and C27–C32) are almost perpendicular to each other, making a dihedral angle of 84.0 (1)°.

One pyrrolidine ring (N1/C1–C4) adopts an envelope conformation, with atom C4 deviating by 0.662 (2) Å from the mean plane through the remaining atoms. The other pyrrolidine ring (N1/C4–C7) adopts a twist conformation with a local twofold axis passing through the N1—C4 bond and atom C6. The puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) for the pyrrolidine rings (N1/C1–C4 and N1/C4–C7) are q_2 = 0.440 (2)/0.424 (2) Å, φ = 330.8 (2)/20.8 (2) and $\Delta_s(C_4)$ = 10.4 (1) and $\Delta_2(C_6)$ = 2.5 (2), respectively.

The molecular conformation is stabilized by weak C—H···O intramolecular interactions. The crystal packing is stabilized by N—H···O and C—H···O hydrogen bonds. Atom N2 acts as a donor to O1 at $(1-x, 1-y, 1-z)$, generating a centrosymmetric dimer of the type $R_2^2(8)$.

Experimental

Isatin (1 mmol), racemic proline (1 mmol), 3,5-dibenzylidene)-1-methylpiperidin-4-one (1 mmol) and 1 g of bentonite clay were mixed thoroughly in a boiling tube which was loosely closed and immersed in a silica-gel bath in a beaker and irradiated in a domestic microwave oven for about 10 min at a maximum power level (600 W) in two 5 min periods with intermittent cooling. The reaction mixture was then cooled to room temperature and tested for the completion of reaction. The product was isolated by column chromatography using *n*-hexane–ethyl acetate in a 5:1 ratio, and was recrystallized (m.p. 441 K) from a hexane–ethyl acetate (4:1) mixture.

Crystal data

$C_{32}H_{31}N_3O_2$	$V = 2643.3$ (2) Å^3
$M_r = 489.60$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 8.7120$ (4) Å	$\mu = 0.08$ mm^{-1}
$b = 20.5711$ (10) Å	$T = 293$ (2) K
$c = 14.9467$ (7) Å	0.25 × 0.24 × 0.22 mm
$\beta = 99.322$ (1)°	

Data collection

Bruker SMART CCD area-detector diffractometer	6227 independent reflections
Absorption correction: none	4738 reflections with $I > 2\sigma(I)$
30223 measured reflections	$R_{\rm int} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$	334 parameters
$wR(F^2) = 0.142$	H-atom parameters constrained
$S = 1.01$	$\Delta\rho_{\rm max} = 0.26$ e Å^{-3}
6227 reflections	$\Delta\rho_{\rm min} = -0.14$ e Å^{-3}

Table 1
Selected bond angles (°).

C4—N1—C1	108.8 (1)	C26—N3—C25	112.5 (1)
C4—N1—C7	107.6 (1)	C26—N3—C24	110.7 (1)
C1—N1—C7	124.1 (1)	C25—N3—C24	111.0 (1)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2	0.98	2.32	2.782 (2)	108
C17—H17···O2	0.93	2.38	2.759 (2)	104
C25—H25*B*···O1	0.97	2.38	2.926 (2)	116
N2—H2···O1$^{\rm i}$	0.86	2.05	2.874 (2)	161
C19—H19···O2$^{\rm ii}$	0.93	2.47	3.226 (2)	139

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x, -y+1, -z$.

All H atoms were refined using a riding model, with C—H = 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for aromatic, C—H = 0.98 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for CH, C—H = 0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for CH_2, C—H = 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for CH_3, and N—H = 0.86 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$.

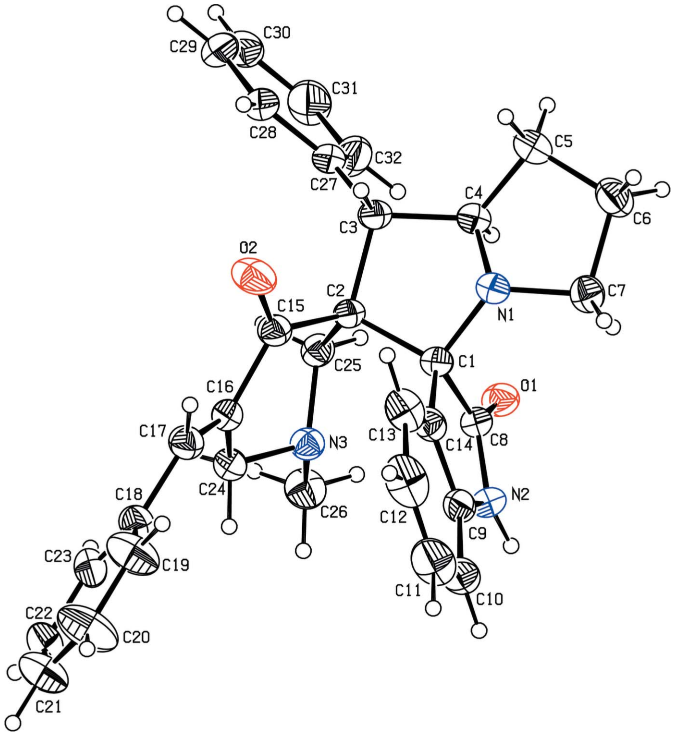


Figure 1
The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

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).

DG thanks the CSIR, India, for the award of a Senior Research Fellowship. The Department of Science & Technology (DST–FIST) and the University Grants Commission (UGC), Government of India, are acknowledged by DV for providing facilities to the department. DV thanks DST, India, for a major research project.

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