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1′-Benzyl-1-(2,4-dimethoxyphenyl)-3-phenoxyspiro[azetidine-2,3′(3*H*)-indole]-2′,4(1*H*)-dione

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.102
Data-to-parameter ratio = 14.5

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

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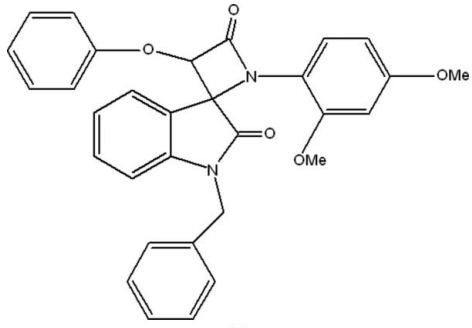
The title compound, $C_{31}H_{26}N_2O_5$, is a compound with a spiro junction between β-lactam and isatin ring systems. Its four- and five-membered rings are nearly planar, with maximum deviations of −0.020 (1) and 0.050 (1) Å for their carbonyl C atoms, respectively. The dihedral angle between these two rings is 86.44 (5)°. The crystal structure is stabilized by intramolecular C—H···O hydrogen-bonding and van der Waals interactions.

Comment

The development of new synthetic methods for the efficient construction of biologically active compounds is an important field in organic chemistry (Alcaide *et al.*, 2001). In this context, the synthesis of diversely substituted monocyclic β-lactams has been of considerable interest to the synthetic community over the past few decades (Jarrahpour *et al.*, 2004*a*,*b*; Jarrahpour & Jahaniani, 2005; Singh, 2003; Gomez-Gallego *et al.*, 2000). The β-lactam skeleton is the key structural unit of the most widely employed β-lactam antibiotics (Durkheimer *et al.*, 1985). As there is a constant need for new drugs displaying broader antibacterial activity and for new β-lactam antibiotics to combat microorganisms that have built up resistance against many traditional drugs (Lopez *et al.*, 2003), continued interest in β-lactams is ensured. In addition to its use in the synthesis of a variety of β-lactam antibiotics, the β-lactam skeleton has been recognized as a useful building block, owing to the utilization of the strain energy associated with the four-membered ring (Alcaide & Almendros, 2001, 2002, 2004; Deshmukh *et al.*, 2004). Consequently, efforts have been made in exploring such new aspects of β-lactam chemistry using enantiomerically pure β-lactams as versatile intermediates for organic syntheses. In particular, spirocyclic β-lactams behave as β-turn mimetics (Alonso *et al.*, 2001), they can act as cholesterol absorption inhibitors (Kambara & Tomioka, 1999) and they are precursors of α,α-disubstituted β-amino acids (Alonso *et al.*, 2002).



(I)

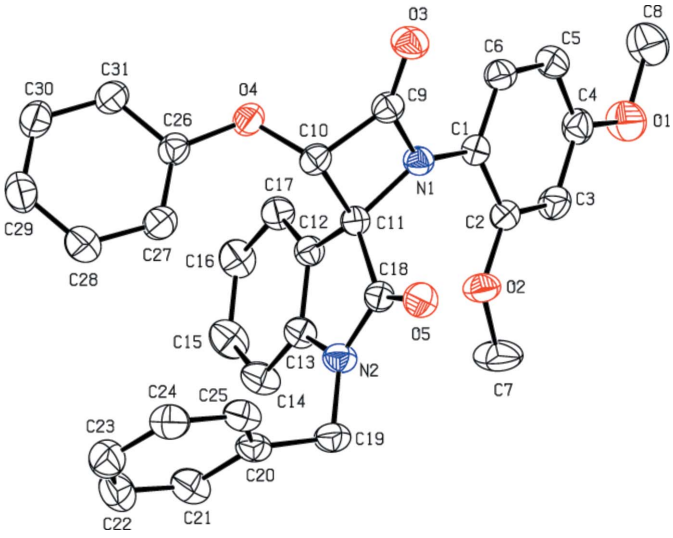


Figure 1
A view of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids. All H atoms have been omitted for clarity.

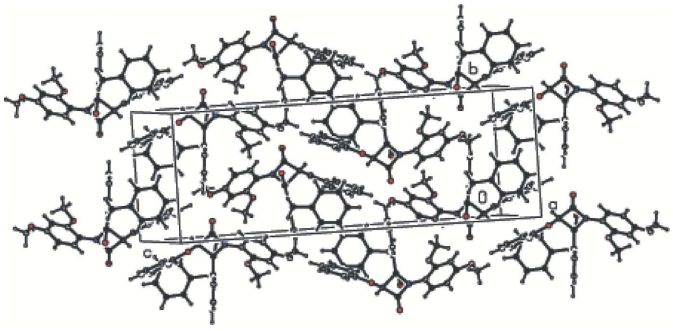


Figure 2
A packing diagram for (I).

The four-membered β-lactam ring of the title compound, (I) (Fig. 1), is nearly planar, with a maximum deviation of 0.020 (1) Å for the carbonyl atom C9. Within the lactam ring, the bond lengths (Table 1) are similar to those observed in previous studies (Ercan *et al.*, 1996*a*,*b*; Kabak *et al.*, 1999). The N1—C9 bond, conjugated with the carbonyl group, is shorter than the N1—C1 and N1—C11 bonds and the C9═O3 bond is longer than the standard value of 1.198 (12) Å given by Allen *et al.* (1987).

The dihedral angles between the ring systems are listed in Table 2. The sum of the bond angles about atom N1 is 357.21°.

The packing of (I), viewed down the *a* axis, is shown in Fig. 2. The crystal structure of (I) is stabilized by intramolecular C—H···O hydrogen bonding (Table 3) and van der Waals interactions.

Experimental

Treatment of *N*-benzyl-3-(2,4-dimethoxyphenylimino)isatin with phenoxyacetyl chloride and triethylamine in dry dichloromethane at 263 K gave the title spiro monocyclic β-lactam (Bhawal *et al.*, 1997). Compound (I) was recrystallized from dichloromethane to give single crystals. The IR spectrum showed the characteristic absorption of β-lactam carbonyl at 1766 cm^{-1} and CO of isatin at 1728 cm^{-1}. The ^{1}H NMR spectrum showed the methoxy H atoms at 3.39 and 3.75 p.p.m., the diastereotopic benzyl H atoms at 4.73 p.p.m (J = 15 Hz) and 5.10 p.p.m (J = 15 Hz), COCHOPh at 5.64 p.p.m, and aromatic H atoms at 6.29–7.93 p.p.m. The ^{13}C NMR spectrum exhibited the following signals: CH_2 benzylic at 44.2, OMe at 55.2 and 55.5, C—OPh at 69.9, spiro C at 86.0, aromatic C at 99.5–159.3, CO of β-lactam at 163.4 and CO of isatin at 173.8 p.p.m. The mass spectrum showed molecular ion at *m*/*e* 506 and the base peak at *m*/*e* 85.

Crystal data

$C_{31}H_{26}N_2O_5$
M_r = 506.54
Monoclinic, $P2_1/c$
a = 9.3547 (7) Å
b = 10.0777 (5) Å
c = 28.193 (2) Å
β = 106.838 (5)°
V = 2543.9 (3) Å^3
Z = 4
D_x = 1.323 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 29809 reflections
θ = 1.5–27.3°
μ = 0.09 mm^{-1}
T = 296 K
Prism, colourless
0.65 × 0.50 × 0.29 mm

Data collection

Stoe IPDS2 diffractometer
ω scans
Absorption correction: none
26901 measured reflections
4995 independent reflections
3963 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 26.0°
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -34 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.102
S = 1.05
4995 reflections
345 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.2246P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C8	1.414 (2)	N1—C1	1.4166 (16)
O2—C7	1.415 (2)	N1—C9	1.3673 (17)
O3—C9	1.2083 (17)	N1—C11	1.4774 (16)
O4—C10	1.4015 (15)	N2—C13	1.4116 (18)
O4—C26	1.3836 (16)	N2—C18	1.3645 (17)
O5—C18	1.2154 (16)	N2—C19	1.4627 (19)
C1—N1—C9	130.19 (11)	C13—N2—C18	110.80 (11)
C1—N1—C11	131.68 (10)	C13—N2—C19	124.66 (11)
C9—N1—C11	95.44 (10)	C18—N2—C19	124.21 (12)

Table 2
Dihedral angles (°) between the planes of the ring systems of (I).

A denotes the ring system N1/C9–C11, *B* C1–C6, *C* C13–C17/C12/C11/C18/N2, *D* C20–C25 and *E* C26–C31.

Rings	*A*	*B*	*C*	*D*	*E*
A		26.81 (5)	84.60 (5)	35.01 (6)	54.04 (5)
B			69.26 (4)	57.04 (5)	51.12 (5)
C				81.68 (4)	64.88 (4)
D					85.54 (5)

Table 3
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O3	0.93	2.47	3.0909 (18)	124
C19—H19A···O5	0.97	2.60	2.9254 (18)	100

All H atoms were positioned geometrically and constrained to an idealized geometry, with C—H distances of 0.93 (aromatic H), 0.96 (methyl H), 0.97 (methylene H) or 0.98 Å (methine H), and with U_{iso}(H) = 1.2U_{eq}(C-aromatic, C-methylene and C-methine) or 1.5U_{eq}(C-methyl).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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