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4-[Bis(2-methyl-1*H*-indol-3-yl)methyl]-1-(4-methoxyphenyl)-3-phenylazetidin-2-one

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In the title compound, $C_{35}H_{31}N_3O_2$, the dihedral angle between the two indole groups is 76.4 (1)°. The molecular structure is stabilized by an intramolecular π–π interaction. The molecular packing in the crystal structure is stabilized by strong N—H···O hydrogen bonds and weak N—H···π interactions.

Comment

The importance and structural diversity of biologically active β-lactam antibiotics, the most widely employed family of antimicrobial agents to date, accounting for 50% of the world's total antibiotic market, led to the development of efficient approaches for the construction of appropriately substituted 2-azetidinones (Alcaide *et al.*, 2000). The minimum structural features believed to be essential for antimicrobial activity in the β-lactam antibiotics have undergone considerable revision, since in recent years several natural monocyclic β-lactams were shown to exhibit high antibacterial activity. Now it seems that the minimum requirement for biological activity is a suitably substituted monocyclic 2-azetidinone ring (Page, 1984). Indole, being an integral part of many natural products of therapeutic importance, possesses potentially reactive sites for a variety of chemical reactions to generate molecular diversity (Farhanullah *et al.*, 2004). In view of its importance and to obtain more detailed information on the structural conformation of the molecule, the crystal structure of the title compound, (I), was determined.

(I)
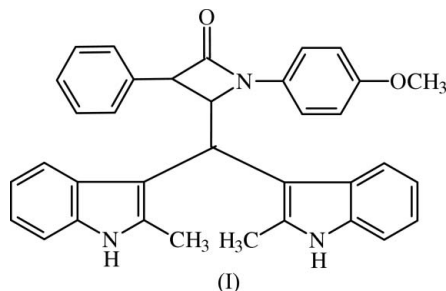
The molecular structure of (I) is illustrated in Fig. 1. Selected geometric parameters are presented in Table 1. The geometry of the β-lactam ring is comparable with a related reported structure (Ercan *et al.*, 1996; Ülkü *et al.*, 1997). The two indole ring systems have bond distances and angles comparable with those reported for other indole derivatives (Karthick *et al.*, 2005; Sonar *et al.*, 2005).

The exocyclic angles around atom C7 show considerable asymmetry, O2—C7—C6 [124.8 (1)°] being wider than O2—C7—C8 [116.0 (1)°]. This difference is due to steric repulsion
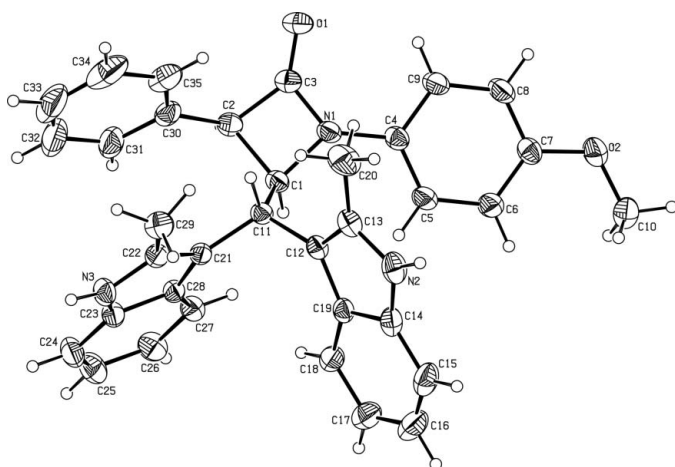
Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.
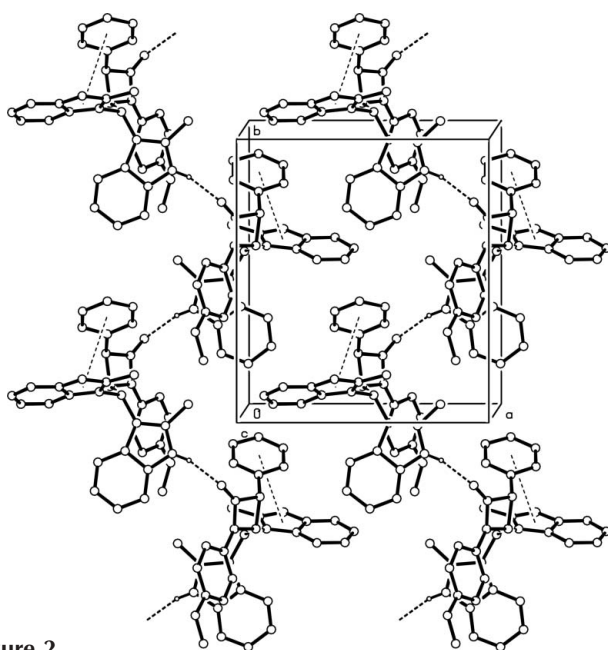

Figure 2
A view of the molecular packing of (I), showing N—H···O, N—H···π and π–π interactions as dashed lines. For the sake of clarity, H atoms not involved in hydrogen bonds have been omitted.

between the benzene ring and the methyl group. The torsion angles C10—O2—C7—C6 [−0.1 (2)°] and C10—O2—C7—C8 [180.0 (2)°] indicate that the methyl group does not deviate significantly from the plane of the attached benzene ring. The two indole groups enclose a dihedral angle of 76.4 (1)°. The phenyl ring and the methoxyphenyl ring enclose a dihedral angle of 55.2 (1)°. The β-lactam ring is planar, with a maximum deviation of 0.109 (1)° for atom O1.

The molecular structure is influenced by a π–π interaction between the five-membered ring (C21/C22/N3/C23/C28) of one indole group and the phenyl ring (C30–C35), with a centroid separation of 3.695 (1) Å (Fig. 2). In the crystal structure, the molecules are linked by N—H···O hydrogen bonds so that a helical structure is formed. In addition,

inversion-related molecules are linked by weak N—H···π interactions: atom H3 is 2.53 Å from the centroid of the five-membered ring composed of atoms C12/C13/N2/C14/C19 at $(1 − x, −y, 1 − z)$, with an N3—H3···centroid angle of 152° and an N3···centroid distance of 3.317 (1) Å.

Experimental

Crystal data

$C_{35}H_{31}N_3O_2$	$D_x = 1.257$ Mg m^{-3}
$M_r = 525.63$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 11212
$a = 11.9751$ (7) Å	reflections
$b = 13.4815$ (8) Å	$\theta = 2.2–23.4°$
$c = 17.2114$ (11) Å	$\mu = 0.08$ mm^{-1}
$\beta = 91.978$ (1)°	$T = 293$ (2) K
$V = 2777.0$ (3) Å^3	Block, colourless
$Z = 4$	$0.26 \times 0.22 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	5325 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.020$
Absorption correction: none	$\theta_{max} = 28.0°$
31001 measured reflections	$h = −15 \rightarrow 15$
6519 independent reflections	$k = −17 \rightarrow 17$
	$l = −22 \rightarrow 22$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.08P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.049$	$+ 0.5932P]$
$wR(F^2) = 0.149$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
6519 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
362 parameters	$\Delta\rho_{min} = −0.22$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.218 (2)	N2—C14	1.372 (2)
N1—C3	1.363 (2)	N2—C13	1.376 (2)
N1—C4	1.411 (2)	N3—C23	1.365 (2)
N1—C1	1.482 (2)	N3—C22	1.371 (2)
C3—N1—C4	132.2 (1)	O2—C7—C6	124.8 (1)
C3—N1—C1	94.5 (1)	C12—C11—C1	116.2 (1)
C4—N1—C1	130.5 (1)	C21—C11—C1	111.2 (1)
O2—C7—C8	116.0 (1)		
C10—O2—C7—C8	180.0 (2)	C2—C1—C11—C12	−156.0 (1)
C10—O2—C7—C6	−0.1 (2)	C2—C1—C11—C21	72.0 (1)

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

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
N2—H2$\cdots$O1^i	0.86	2.10	2.907 (2)	157

Symmetry code: (i) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$.

H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H distances of 0.93–0.98 Å and N—H distances of 0.86 Å, and with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C,N)$ for the other H atoms. All methyl groups were allowed to rotate but not to tip.

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: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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