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Ethyl 2-(1*H*-indol-3-yl)-5-[1-(4-methoxyphenyl)-4-oxo-3-phenylazetidin-2-yl]-4-nitro-3-(*p*-tolyl)-pyrrolidine-2-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.053
wR factor = 0.147
Data-to-parameter ratio = 9.3

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

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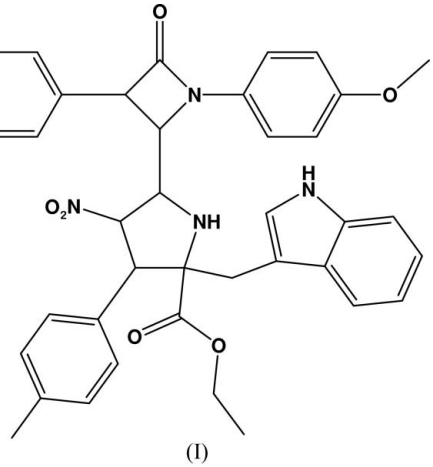
In the title molecule, $C_{39}H_{38}N_4O_6$, the pyrrolidine ring adopts a twist conformation. The crystal packing is stabilized by N—H···O and C—H···O intermolecular interactions.

Comment

The β-lactam ring plays a key role in the most widely employed class of antimicrobial agents. The newer β-lactam antibiotics can be highly effective in combating infections caused by β-lactamase-producing organisms. Therefore, much effort has been extended in recent years to prepare new structural types having a β-lactam ring as a common feature, which will overcome the defence mechanisms of the bacteria (Alcaide *et al.*, 2003). As the β-lactam derivative is of much importance, we have underataken the X-ray crystal structure determination of the title compound, (I).



(I)

The sums of the bond angles around N3 (359.9) and N4 (359.6) indicate that they are sp^2-hybridized. The internal angles in the β-lactam ring vary from 84.8 (2) to 95.1 (3)°. The azetidin-2-one group is planar and the attached C26–C31 phenyl and C33–C38 benzene rings are twisted away by 71.4 (2) and 31.7 (2)°, respectively. The dihedral angle between the C26–C31 and C33–C38 rings is 40.5 (1)°. The methoxy group at C36 is twisted away from the attached ring, with a torsion angle C35—C36—O6—C39 of −161.0 (5)°.

The indole ring system is planar to within ±0.014 (3) Å. The pyrrolidine ring adopts a twist conformation, with a pseudo-twofold axis passing through atom C2 and the C4—N1 bond; the puckering parameters (Cremer & Pople, 1975) and the smallest displacement asymmetry parameters (Nardelli, 1983) are q_2 = 0.371 (3) Å, φ = 334.7 (4)° and $\Delta_s(C_2)$ = 6.6 (2)°.

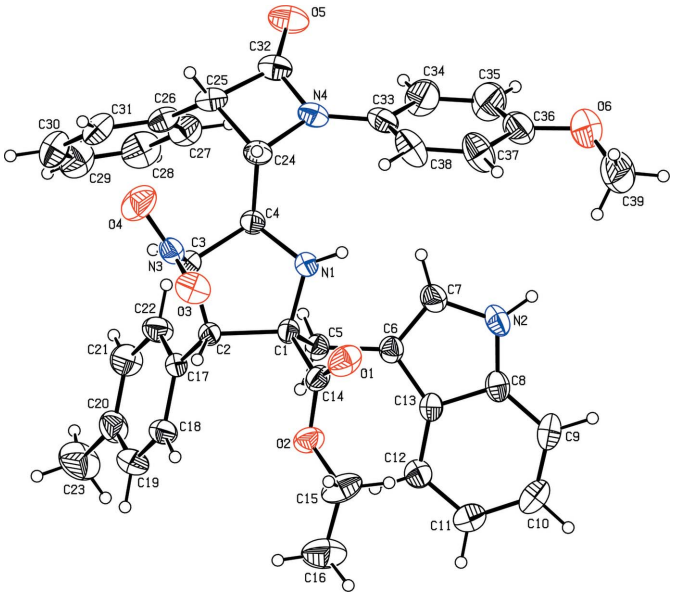


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

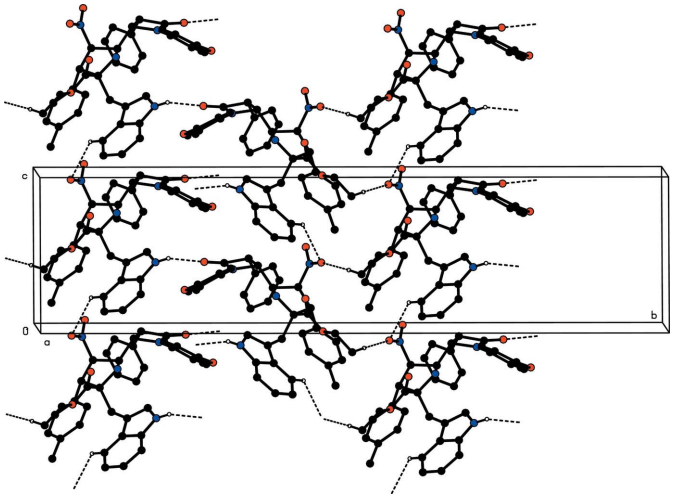


Figure 2
The molecular packing of (I), viewed approximately down the *a* axis. For clarity, H atoms not involved in the hydrogen bonds (dashed lines) have been omitted.

The molecular structure is stabilized by a weak C2—H2···O3 interaction. The crystal packing is stabilized by N—H···O and C—H···O intermolecular interactions (Table 2). Atom N2 acts as a donor to O5 at $(\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z)$, generating a $C(12)$ chain. Atom C12 acts as a donor to O3 at $(x, y, -1+z)$, generating a $C(10)$ chain along the *c* axis (Fig. 2). Atom C16 acts as a donor to O3 at $(x, -y, -\frac{1}{2}+z)$, generating a $C(10)$ chain.

Experimental

4-Formylazetidin-2-one (1 mmol) was treated with tryptophan ethyl ester hydrochloride (1 mmol) in the presence of Et_3N (2.5 mmol) and anhydrous $MgSO_4$ (10 g) in dry dichloromethane (10 ml) at room temperature afforded the imine, (*E*)-ethyl-2-[1-(4-methoxyphenyl)-4-oxo-3-phenylazetin-2-yl]methyleneamino)-3-(1*H*-indol-3-yl)propanoate. The imine (1 mmol) was then stirred with silver(I) acetate (1catalytic amount) and *p*-methylnitrostyrene (1 mmol) in the presence of Et_3N (1.2 mmol) and molecular sieves in dry toluene (30 ml) at room temperature for 12 h. The reaction mixture was filtered through a plug of Celite. The solvent was evaporated under reduced pressure and the residue was subjected to column chromatography on silica gel (100–200 mesh), eluting with hexane–ethyl acetate (7:3), to give the title compound. The compound was recrystallized from ethyl acetate by slow evaporation.

Crystal data

$C_{39}H_{38}N_4O_6$
M_r = 658.73
Monoclinic, *Cc*
a = 11.2689 (8) Å
b = 35.612 (2) Å
c = 8.8848 (6) Å
β = 99.785 (1)°
V = 3513.7 (4) Å^3
Z = 4
Mo *K*α radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.27 × 0.24 × 0.23 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
20233 measured reflections
4167 independent reflections
3771 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.147
S = 1.13
4167 reflections
446 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.459 (3)	C8—N2	1.349 (4)
C3—N3	1.511 (4)	C14—O1	1.201 (4)
C4—N1	1.452 (3)	C24—N4	1.477 (4)
C7—N2	1.371 (4)	C32—O5	1.207 (4)
N4—C24—C25	86.7 (2)	O3—N3—C3	119.2 (3)
C32—C25—C24	84.8 (2)	C32—N4—C33	131.2 (3)
O4—N3—O3	123.0 (3)	C32—N4—C24	95.1 (3)
O4—N3—C3	117.7 (3)	C33—N4—C24	133.3 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O3	0.98	2.29	2.742 (4)	107
N2—H2*A*···O5^{i}	0.86	2.05	2.829 (4)	149
C12—H12···O3^{ii}	0.93	2.57	3.216 (4)	127
C16—H16*A*···O3^{iii}	0.96	2.55	3.488 (7)	167

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

H atoms were positioned geometrically and allowed to ride on their parent atoms, with N—H = 0.86 Å, C—H = 0.93–0.98 Å and U_{iso}(H) = 1.5U_{eq}(methyl C) and 1.2U_{eq}(C,N). In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

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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