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4-[3-Benzoyl-4-(4-chlorophenyl)-1-methylpyrrolidin-2-yl]-1-(4-methoxyphenyl)-3-phenylazetidin-2-one

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In the title compound, $C_{34}H_{31}ClN_2O_3$, the pyrrolidine ring adopts an envelope conformation. The molecular conformation is stabilized only by an intramolecular C—H···O interaction.

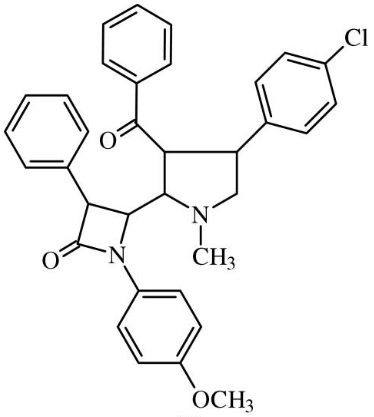
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.058
wR factor = 0.149
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.

Comment

β-Lactam antibiotics account for 50% of the total antibiotic market of the world. The extensive use of common β-lactam antibiotics, such as penicillins and cephalosporins, in medicine has resulted in an increasing number of resistant bacteria through mutation and β-lactamase gene transfer. There has, therefore, been much effort expended in recent years to prepare new structural types having a 2-azetidinone ring as a common feature, which will overcome the defence mechanisms of the bacteria (Alcaide *et al.*, 2003). In recent years, several natural monocyclic β-lactams were shown to exhibit high antibacterial activity, suggesting that a suitably substituted monocyclic 2-azetidinone ring might perhaps be the minimum requirement for biological activity (Page, 1984). In view of its importance and to obtain more detailed information of the structure and conformation of the molecule, the crystal structure analysis of (I) was undertaken.



(I)

The molecular structure of (I) is illustrated in Fig. 1. Selected geometric parameters are presented in Table 1. The Cl—C bond length agrees well with the reported mean value of 1.739 (10) Å (Allen *et al.*, 1987). The β-lactam ring is planar, with bond distances and angles comparable with values reported for other β-lactam derivatives (Selvanayagam *et al.*, 2005; Ülkü *et al.*, 1997).

The sum of the angles at N2 of the pyrrolidine ring [330.0°] is in accordance with sp^3 hybridization. The methyl group is attached equatorially to the pyrrolidine ring. The torsion

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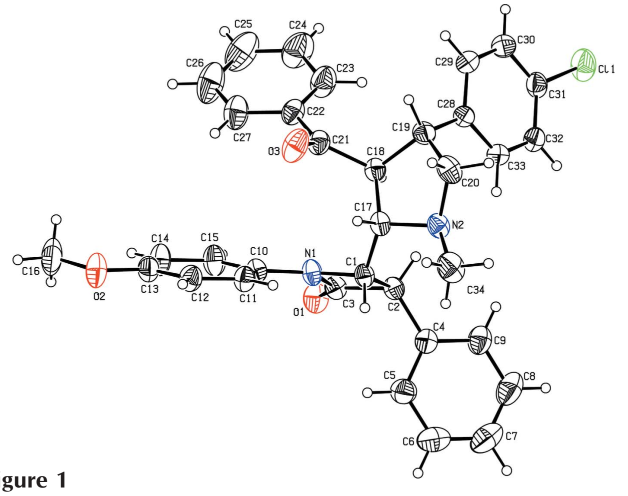

Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

angles C14—C13—O2—C16 [−5.4 (4)°] and C12—C13—O2—C16 [174.9 (2)°] indicate that the methoxy group does not deviate significantly from the plane of the benzene ring to which it is attached.

The pyrrolidine ring adopts an envelope conformation with puckering parameters q_2 = 0.434 (2) Å and φ = 176.7 (2)° (Cremer & Pople, 1975). Atom N2 deviates by 0.639 (2) Å from the least-squares plane through the remaining four atoms (C17–C20). The phenyl ring and the methoxyphenyl ring make dihedral angles of 89.2 (1) and 21.5 (1)°, respectively, with the *β*-lactam ring. The molecular conformation is stabilized by a C—H···O intramolecular interaction (Table 2).

Experimental

A solution of *cis*-4-formyl-2-azetidinone (1 mmol), sarcosine (1 mmol) and chlorochalcone (1 mmol) was refluxed in toluene (15 ml) using a Dean–Stark apparatus. The completion of the reaction was evidenced by thin-layer chromatography. The solvent was then removed in a vacuum. The crude product was subjected to column chromatography using petroleum ether–ethyl acetate (4:1) to afford the title compound. The compound was recrystallized from methanol to obtain diffraction quality crystals.

Crystal data

$C_{34}H_{31}ClN_2O_3$
M_r = 551.06
Monoclinic, $P2_1/n$
a = 11.3180 (8) Å
b = 11.6376 (8) Å
c = 23.0980 (16) Å
β = 103.000 (1)°
V = 2964.4 (4) Å^3
Z = 4
D_x = 1.235 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4958 reflections
θ = 2.3–27.2°
μ = 0.17 mm^{-1}
T = 293 (2) K
Block, colourless
0.26 × 0.24 × 0.20 mm

Data collection

Bruker SMART APEX area-detector diffractometer
ω scans
Absorption correction: none
17499 measured reflections
6766 independent reflections
4748 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.019
$\theta_{\max}$ = 28.0°
$h = -14 \rightarrow 14$
$k = -15 \rightarrow 10$
$l = -29 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.149
S = 1.03
6766 reflections
363 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0679P)^2 + 0.61P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.28 e Å^{-3}
$\Delta\rho_{\min}$ = −0.17 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C31	1.743 (2)	N2—C34	1.460 (3)
O1—C3	1.202 (2)	N2—C17	1.462 (2)
O3—C21	1.206 (2)	C1—C2	1.550 (2)
N1—C3	1.369 (2)	C2—C3	1.536 (2)
N1—C10	1.409 (2)	C17—C18	1.529 (3)
N1—C1	1.479 (2)	C18—C19	1.576 (2)
N2—C20	1.456 (3)	C19—C20	1.523 (3)
C20—N2—C34	113.1 (2)	C34—N2—C17	114.0 (2)
C20—N2—C17	102.9 (2)		
C1—C2—C4—C5	50.3 (2)	C16—O2—C13—C12	174.9 (2)
C1—N1—C10—C11	−9.5 (3)	C34—N2—C17—C18	167.0 (2)
C16—O2—C13—C14	−5.4 (4)	C34—N2—C20—C19	−169.7 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C17—H17···O3	0.98	2.39	2.827 (2)	106

The H atoms were positioned geometrically and were treated as riding on their parent atoms, with C—H distances in the range 0.93–0.98 Å, and with $U_{\rm iso}$= 1.5$U_{\rm eq}$(C) for methyl H and 1.2$U_{\rm eq}$(C) for other H atoms.

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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References

Alcaide, B., Almendros, P., Alonso, J. M. & Redondo, M. C. (2003). *J. Org. Chem.* **68**, 1426–1432.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, S1–19.
Bruker (2001). *SAINT* (Version 6.28a) and *SMART* (Version 5.625). Bruker AXS Inc., Madison, Wisconsin, USA.
Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Page, M. I. (1984). *Acc. Chem. Res.* **17**, 144–151.
Selvanayagam, S., Velmurugan, D., Ravikumar, K., Sridhar, B. & Ramesh, E. (2005). *Acta Cryst.* E**61**, o3386–o3388.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Ülkü, D., Ercan, F. & Güner, V. (1997). *Acta Cryst.* C**53**, 1945–1947.