Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Benzyl 2-(5-methyl-2-pyridylaminocarbonyl)-pyrrolidine-2-carboxylate

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Received 12 August 2006
Accepted 12 August 2006

In the title compound, $C_{19}H_{21}N_3O_3$, the pyrrolidine ring possesses an envelope conformation. The crystal packing is stabilized by N—H···O and C—H···O hydrogen bonds.

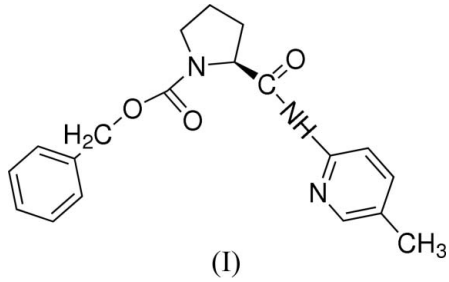
Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.002 Å
R factor = 0.028
wR factor = 0.072
Data-to-parameter ratio = 9.8

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

Comment

The title compound, (I), is an important inhibitor of peptidyl deformylase (Patel *et al.*, 2002). Its crystal structure is reported here.



(I)

The molecular structure of (I) is shown in Fig. 1. Bond lengths and angles in (I) are normal. The pyrrolidine ring possesses an envelope conformation. The dihedral angle between the C6-benzene and O1/O2/N1/C8 planes is 47.51 (7)° and that between the N3-pyridine and O3/N2/C12/C13 planes is 6.74 (6)°. The crystal packing is stabilized by N—H···O and C—H···O hydrogen bonds (Table 1).

Experimental

N-Carbobenzyloxy-L-proline (2.0 g, 8.0 mmol) and triethylamine (0.81 g, 8.0 mmol) were dissolved in tetrahydrofuran (30 ml). To the solution was added dropwise ethyl chloroformate (0.88 g, 8.0 mmol) at 273 K. The solution was stirred for 30 min, and then 5-methylpyridin-2-amine (8.0 mmol) was added over a period of 15 min. The resulting solution was stirred at 273 K for 1 h and at room temperature for a further 16 h, and was then refluxed for 3 h. After it had been cooled to room temperature, the solution was diluted with ethyl acetate. After filtration and removal of the solvent under reduced pressure, the residue was purified through column chromatography on silica gel to give compound (I). Colourless single crystals of (I) were obtained by recrystallization from an ethanol solution.

Crystal data

$C_{19}H_{21}N_3O_3$
M_r = 339.39
Orthorhombic, $P2_12_12_1$
a = 9.2716 (3) Å
b = 11.4256 (3) Å
c = 16.4220 (4) Å
V = 1739.64 (8) Å^3
Z = 4
D_x = 1.296 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 153 (2) K
Block, colourless
0.60 × 0.39 × 0.30 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: none
17024 measured reflections
2272 independent reflections
2217 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 27.5^{\circ}$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.072$
$S = 1.03$
2272 reflections
232 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0439P)^2 + 0.268P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0120 (17)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2N···O2^{i}	0.86 (2)	2.09 (2)	2.9273 (15)	162.5 (17)
C3—H3···O3^{ii}	0.95	2.59	3.3869 (18)	142
C12—H12···O2^{i}	1.00	2.58	3.3024 (16)	129
C16—H16···O3^{iii}	0.95	2.54	3.4664 (17)	165

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

Atom H2N was located in a difference Fourier map and refined isotropically. Other H atoms were placed in calculated positions, with C—H = 0.95–1.00 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C). The torsion angle for the methyl group was refined to fit the electron density. In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

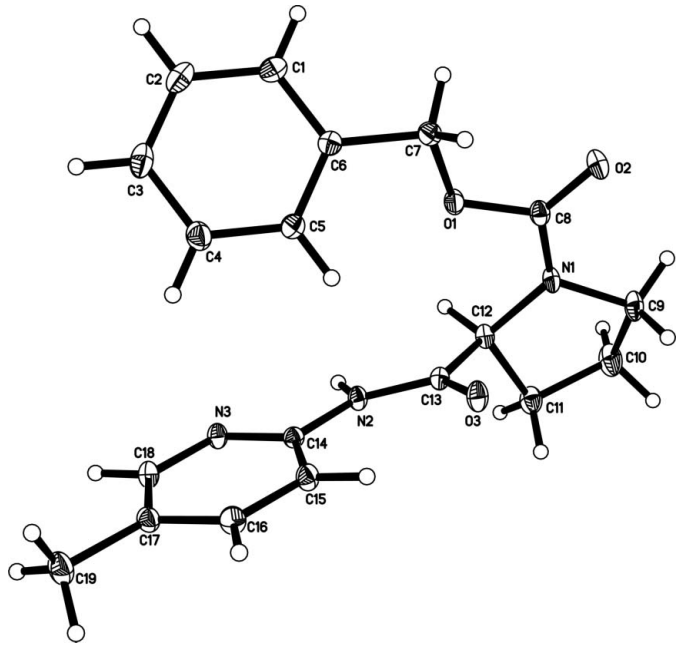


Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

The author thanks the Centre for Testing and Analysis, Chengdu Branch, Chinese Academy of Sciences, for analytical support.

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