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(*S*)-3-Hydroxy-1-(*N*-*p*-nitrobenzyloxycarbonylacetimidoyl)pyrrolidine

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The crystal structure of the title compound, $C_{14}H_{17}N_3O_5$, is stabilized by intermolecular O—H···N hydrogen bonds.

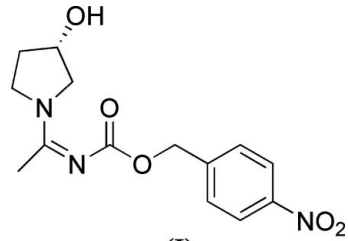
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.108
Data-to-parameter ratio = 8.4

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

Comment

Carbapenem antibiotics represent a promising set of compounds which exhibit excellent antiviral properties (Albers-Schonberg *et al.*, 1978; Kondo *et al.*, 1997). To date, several carbapenem antibiotics have appeared on the market, *e.g.* imipenem, panipenem, meropenem, biapenem, ertapenem and doripenem. During our research on panipenem (Miyadera *et al.*, 1983), we synthesized and crystallized the title intermediate, (I), and present its crystal structure here.



(I)

The hydroxyl group is in an axial position of the pyrrolidine ring. The crystal structure is stabilized by intermolecular O—H···N hydrogen bonds (Table 1 and Fig. 2).

Experimental

The title compound was synthesized according to the method of Miyadera *et al.* (1995). Solvents were chromatographically pure, and triethylamine was dried using a molecular sieve before use. 3-

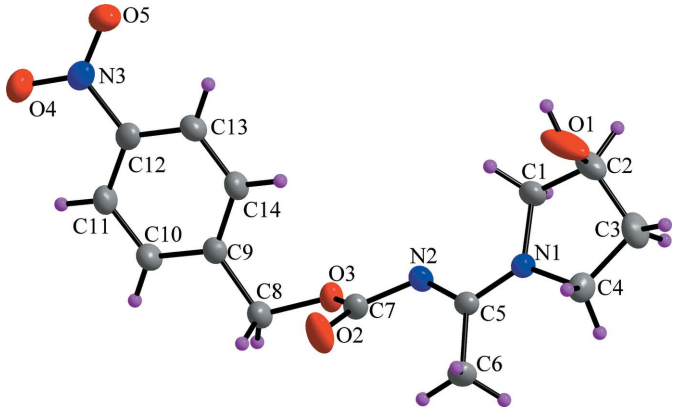


Figure 1
The molecular structure of the title compound, showing 30% probability displacement ellipsoids (spheres of arbitrary radius for the H atoms).

Hydroxypyrrolidine hydrochloride, ethyl acetimidate hydrochloride and *p*-nitrobenzyloxycarbonyl chloride were dried under vacuum for 4 h before use. Other chemicals were used as received without further purification. To a suspension of 3-hydroxypyrrolidine hydrochloride (1.23 g) in ethanol (10 ml) was added triethylamine (1.4 ml), followed by ethyl acetimidate hydrochloride (1.23 g). The mixture was then stirred at room temperature for 1 h. At the end of this time, the solvent was distilled off and dichloromethane (10 ml) was added. The mixture was ice-cooled, and then *p*-nitrobenzyloxycarbonyl chloride (2.2 g) was added. Triethylamine (1.4 ml) was added dropwise and the whole mixture was stirred for 1 h. The mixture was then extracted with water, washed with brine and dried. The solvent was distilled off to obtain the title compound as a light-yellow solid (51% yield). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution in ethyl acetate (m.p. 391–393 K).

Crystal data

$C_{14}H_{17}N_3O_5$
M_r = 307.31
Monoclinic, $P2_1$
a = 7.2123 (5) Å
b = 9.2723 (6) Å
c = 10.9592 (7) Å
β = 92.0580 (10)°
V = 732.42 (8) Å^3
Z = 2
D_x = 1.393 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (2) K
Block, colourless
0.48 × 0.45 × 0.42 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.950, T_{max} = 0.956
4426 measured reflections
1703 independent reflections
1575 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.108
S = 1.05
1703 reflections
202 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 0.176P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.155 (13)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2^{i}	0.82	2.27	2.879 (3)	132

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

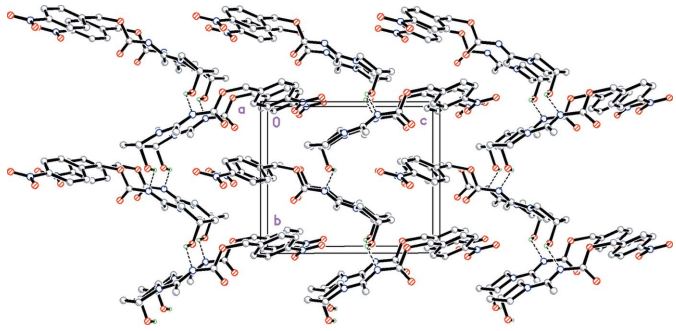


Figure 2
The crystal packing of the title compound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

All H atoms were placed in geometrically idealized positions, with C—H = 0.96 Å, and refined as riding, with U_{iso}(H) = 1.5U_{eq}(C). In addition, the torsion angles about the methyl and hydroxyl groups were refined. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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