Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(4*S*)-Benzyl 4-methyl-5-oxo-1,3-oxazolidine-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.007 Å
R factor = 0.056
wR factor = 0.139
Data-to-parameter ratio = 8.1

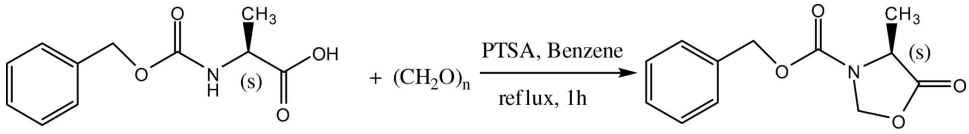
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 30 November 2006
Accepted 6 December 2006

In the title compound, $C_{12}H_{13}NO_4$, the phenyl and oxazolidine rings make a dihedral angle of 65.0 (1)°. Weak C—H···O hydrogen-bonding interactions lead to the formation of a chain parallel to the *a* axis.

Comment

The title compound, (I), has been used to synthesize dipeptides and *N*-methyl-α-amino acids (Dorow & Gingrich, 1999). It can also be used in the synthesis of a series of biologically active molecules.



(I)

The phenyl and the oxazolidine rings make a dihedral angle of 65.0 (1)° (Fig. 1). The absolute configuration (*S*) of the stereocentre C3 remains unchanged during the synthetic procedure. The occurrence of weak C—H···O hydrogen-bonding interactions leads to the formation of a linear chain parallel to the *a* axis (Table 1). Bond lengths and angles are in agreement with values reported in the literature (Thompson *et al.*, 1999).

Experimental

N-Cbz-L-alanine (6.69 g, 3 mmol), paraformaldehyde (1.8 g, 6 mmol) and 4-methylbenzenesulfonic acid (PTSA, 0.35 g, 1.8 mmol) were dissolved in benzene (250 ml). The solution was refluxed for 1 h with a Dean–Stark trap. After cooling to room temperature, the mixture was washed with 0.3 *M* aqueous K_2CO_3 solution, water, and saturated aqueous NaCl solution, then dried over Mg_2SO_4 (Reddy *et al.*, 2000). A white solid was obtained after evaporation of the solvent. Single crystals were obtained by recrystallization from ethanol.

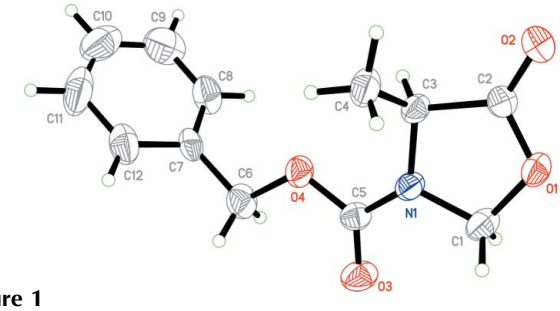


Figure 1
The molecular structure of the title compound with the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

Crystal data

$C_{12}H_{13}NO_4$
M_r = 235.23
Orthorhombic, $P2_12_12_1$
a = 6.1357 (19) Å
b = 8.568 (3) Å
c = 22.758 (7) Å
V = 1196.4 (7) Å^3
Z = 4
D_x = 1.306 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 273 (2) K
Chunk, colorless
0.48 × 0.32 × 0.21 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.954, T_{max} = 0.980
5764 measured reflections
1258 independent reflections
1209 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.139
S = 1.20
1258 reflections
155 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.3568P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O2^{i}	0.97	2.49	3.319 (5)	144

Symmetry code: (i) $x+1, y, z$.

All H atoms were placed in geometrically idealized positions and treated as riding on their parent atoms, with C—H = 0.93 (aromatic), 0.98 (CH), 0.97 (CH_2) or 0.96Å (CH_3), and U_{iso}(H) = 1.2U_{eq}(aro-eq(aromatic, CH and CH_2) or 1.5U_{eq}(methyl C). In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration was assumed from the synthesis.

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: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The project was supported by the National Natural Science Foundation of China (No. 20572061) and we also thank the Key Laboratory for Physical Chemistry of the Solid Surface for providing the X-ray diffraction facilities.

References

Bruker (2001). *SAINT* (Version 6.22), *SMART* (Version 5.625) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Dorow, R. L. & Gingrich, D. E. (1999). *Tetrahedron Lett.* **40**, 467–470.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Reddy, G. V., Rao, G. V., Sreevani, V. & Iyengar, D. S. (2000). *Tetrahedron Lett.* **41**, 953–954.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Thompson, M. J., Mekhalfia, A., Hornby, D. P. & Blackburn, G. M. (1999). *J. Org. Chem.* **64**, 7467–7473.