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A short depsipeptide: *tert*-butoxycarbonyl-L-alanyl-L-lactic acid benzyl ester (Boc-L-Ala-L-Lac-OBzl)

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The title compound, $C_{18}H_{25}N_1O_6$, crystallizes in an extended sheet-like conformation. In the crystal structure, independent molecules are linked by an $N—H\cdots O=C$ hydrogen bond, yielding an infinite arrangement.

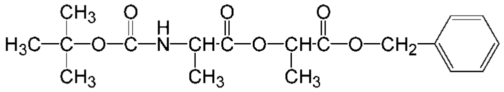
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.009 Å
R factor = 0.070
wR factor = 0.182
Data-to-parameter ratio = 9.1

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 a key starting material in the production of bioabsorbable polymers, such as -(Ala-Ala-Lac)$_n$- (Katakai & Goodman, 1982). Similar compounds, containing polydepsipeptide and oligodepsipeptide sequences, have been extensively studied in our laboratory (Katakai & Goodman, 1982; Katakai, 1988; Katakai *et al.*, 1995, 2004; Ohyama *et al.*, 2000, 2001; Yasuno *et al.*, 2001; Yoshida *et al.*, 1990).



Boc-L-Ala-L-Lac-OBzl

(I)

The molecular structure of (I) is shown in Fig. 1; the bond lengths and angles are unexceptional. The molecule has an extended sheet-like conformation. As shown in Fig. 2, independent molecules are linked by an $NH\cdots O=C$ interaction, yielding an infinite arrangement.

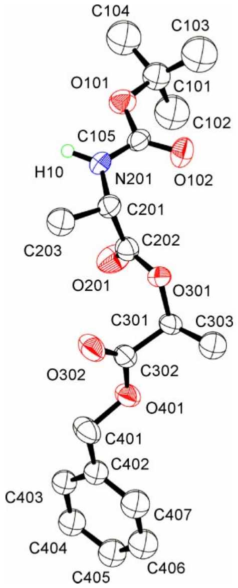


Figure 1
The structure of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. All H atoms have been omitted, except for those involved in hydrogen bonding.

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The melting point of (I) is 356–359 K. Relatively low values are often observed for short sequences, especially those containing a hydroxyacid residue (Oku *et al.*, 2003, 2003*a*,*b*). In these cases, the number of hydrogen bonds definitely influences the melting temperature.

Experimental

The title compound, (I), was isolated from a reaction mixture of a liquid-phase peptide synthesis (Ohyama *et al.*, 2000, 2001). Crystals of the title compound were successfully grown by the slow evaporation of an ethyl acetate solution. Analytical data (melting point, ^{1}H NMR, ESI–MS, and $[\alpha]_D^{20}$) are in accordance with the expected structure; m.p. 356–359 K, $[\alpha]_D^{20} = -44.8°$ (*c* 0.1, chloroform).

Crystal data

$C_{18}H_{25}NO_6$
$M_r = 351.40$
Monoclinic, $P2_1$
$a = 10.365$ (6) Å
$b = 8.827$ (5) Å
$c = 11.001$ (6) Å
$\beta = 104.93$ (2)°
$V = 972.4$ (9) Å^3
$Z = 2$
$D_x = 1.200$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 4037 reflections
$\theta = 4.2$–67.5°
$\mu = 0.75$ mm^{-1}
$T = 173.1$ K
Prism, colorless
0.40 × 0.30 × 0.05 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.789$, $T_{\max} = 0.963$
15424 measured reflections
1747 independent reflections
1624 reflections with $F^2 > 2\sigma(F^2)$
$R_{\rm int} = 0.029$
$\theta_{\max} = 68.2°$
$h = -12 \rightarrow 12$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.070$
$wR(F^2) = 0.182$
$S = 0.94$
1747 reflections
191 parameters
All H-atom parameters constrained
$w = 1/[0.005F_o^2 + 20\sigma(F_o^2) + 0.5]/(4F_o^2)$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.33$ e Å^{-3}

Table 1
Selected torsion angles (°).

O101—C105—N201—C201	177.4 (4)	C202—O301—C301—C302	−69.3 (5)
C105—N201—C201—C202	−66.9 (6)	O301—C301—C302—O401	162.7 (5)
N201—C201—C202—O301	145.9 (5)	C301—C302—O401—C401	174.9 (4)
C201—C202—O301—C301	166.4 (4)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N201—H10···O302$^{\rm i}$	0.95	2.07	2.946 (6)	153

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

The *tert*-butyl C atoms, those of the benzene ring, and C203 and C303 were refined isotropically; all other non-H atoms were refined anisotropically. This procedure was adopted because of the limited number of observed reflections. H atoms were positioned geometrically and refined using a riding model, with C—H and N—H distances of 0.95 Å. $U_{\rm iso}$(H) values were constrained to be $1.2U_{\rm eq}$ of the carrier atom. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged. The absolute configuration was confirmed from the analytical data, α_D, of the compound.

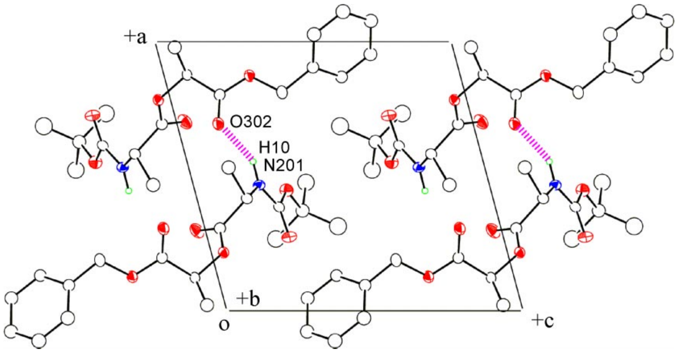

Figure 2
A packing diagram of (I), projected down the *b* axis. Hydrogen bonds are indicated as dashed lines.

Data collection: *RAPID-AUTO* (Rigaku/MSC and Rigaku, 2003); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC and Rigaku, 2003); program(s) used to solve structure: *SIR*2002 (Burla *et al.*, 2003); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 1996); molecular graphics: *ORTEP* (Johnson, 1965); software used to prepare material for publication: *CrystalStructure*.

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