organic papers

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Key indicators

Single-crystal X-ray study
$T = 120$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.117
Data-to-parameter ratio = 9.5

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

4(R)-(N-Benzoylamino)-5(R)-methyl-tetrahydrofuran-2-one: an L-β-threonine analogue

The title compound, $C_{12}H_{13}NO_3$, was synthesized from L-β-threonine, and the lactone ring was formed by nucleophilic reaction of the β-hydroxyl group in the presence of a base. The methyl and amino groups are located at *cis* positions, but the puckering of the lactone ring reduces their steric hindrance. The benzoyl π-electrons and amide bond were expected to conjugate to each other, but their least-square planes are inclined at 26.3 (1)°. The hydrogen bonds between the amide bonds extend along the *b* axis and stabilize the molecular packing.

Comment

The title compound, (I), is a lactone synthesized from *O*-benzyl-L-β-threonine methyl ester, (II). Hydrogenation of (II) deprotects the benzyl group. The resulting alcoholic hydroxyl group is a nucleophile. Lactonization with the carboxyl C atom is caused by bases, with elimination of the methyl ester. Similar reactions have been known for Ser and Thr (Sheehan *et al.*, 1959), but the elongated backbone of the β-amino acid promotes lactonization. This method conveniently yields amino-lactones, whose chirality is derived from threonines. Moreover, the extraction and purification procedures are easy for benzoyl lactones. In this paper, the structure of (I), derived from L-threonine, is reported.
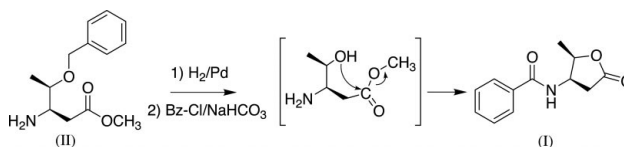
The structure of (I) is shown in Fig. 1. The absolute configuration is established from the L(R)-chirality of atom C1A. The configuration of atom C2B is also R, and the methyl and amino groups are located *cis* with respect to one another [N1—C1A—C2B—C3G = 30.5 (3)°; Table 1]. This form may lead to contacts between atoms N1 and C3G, but the puckering of the lactone ring reduces short contacts [N1···C3G = 2.877 (3) Å]. Steric hindrance is observed between the H atoms on N1 (H6) and C12 (H1) (H6···H1 = 2.152 Å), and the phenyl ring is tilted about the amide bond [C12—C11—C17—N1 = −26.4 (3)°]. The angle between the least-square planes through the phenyl ring (C11–C16) and the amide group (C11, C17, O17, N1 and C1A) is 26.3 (1)°, essentially equal to the torsion angle about the C11—C17 bond. Atom N1 is hydrogen bonded to atom O17 of an adjacent molecule [N1···O17 = 2.973 (3) Å; Table 2]. This linkage extends along the *b* axis, as shown in Fig. 2, and stabilizes the molecular packing.

Experimental

O-Benzyl-β-threonine methyl ester, (II), was synthesized as described previously (Seebach *et al.*, 1996; Gopi *et al.*, 2002). Compound (II) (7.1 g, 32 mmol) was dissolved in methanol (200 ml), and an aqueous suspension of Pd/C (6 g in 100 ml) was added. After catalytic hydrogenation, the solution was filtered and concentrated under reduced pressure. The residue was dissolved in an aqueous 8% NaHCO$_3$ solution (200 ml) and benzoyl chloride (13.6 g) was added with diethyl ether (100 ml). After 5–6 h, the diethyl ether phase was extracted and the aqueous phase was washed three times with ethyl acetate (100 ml). The organic phases were combined and concentrated after drying with Na$_2$SO$_4$. Recrystallization using ethyl acetate–diethyl ether yielded (I) (6.13 g, 87%). ^{1}H NMR (500 MHz, CDCl$_3$): δ 1.41 (3H, *d*, *J* = 6.53 Hz, C3*G*—H$_3$), 2.61 (1H, *d*, *d*, *J* = 18.00, 1.84 Hz, C4*B*—H$_2$), 4.85 (1H, *q*, *d*, *J* = 6.53, 5.17Hz, C2*B*—H), 5.08 (1H, *t*, *d*, *d*, *J* = 8.18, 5.17, 1.84 Hz, C1*A*—H), 7.31 (1H, *br*, N1—H), 7.45 (2H, *m*, C$_6$H$_5$), 7.52 (1H, *m*, C$_6$H$_5$), 7.86 (2H, *m*, C$_6$H$_5$).

Crystal data

C$_{12}$H$_{13}$NO$_3$	$D_x = 1.364$ Mg m^{-3}
$M_r = 219.23$	Mo $K\alpha$ radiation
Monoclinic, $P2_1$	Cell parameters from 2050
$a = 8.521$ (1) Å	reflections
$b = 5.1143$ (6) Å	$\theta = 2.4–28.3°$
$c = 12.401$ (2) Å	$\mu = 0.10$ mm^{-1}
$\beta = 99.098$ (2)°	$T = 120.0$ (2) K
$V = 533.6$ (1) Å^3	Needle, colourless
$Z = 2$	$0.50 \times 0.06 \times 0.03$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1385 independent reflections
	1339 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.024$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 27.9°$
	$h = -10 \rightarrow 10$
$T_{min} = 0.843$, $T_{max} = 0.997$	$k = -6 \rightarrow 6$
4798 measured reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0819P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.043$	$+ 0.0481P]$
$wR(F^2) = 0.118$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.12$	$(\Delta/\sigma)_{max} < 0.001$
1385 reflections	$\Delta\rho_{max} = 0.40$ e Å^{-3}
146 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (°).

C12—C11—C17—N1	−26.4 (3)	C1*A*—C2*B*—O3*G*—C5	−18.2 (2)
C11—C17—N1—C1*A*	178.53 (18)	N1—C1*A*—C4*B*—C5	−150.28 (18)
N1—C1*A*—C2*B*—O3*G*	150.75 (17)	C2*B*—C1*A*—C4*B*—C5	−26.4 (2)
C4*B*—C1*A*—C2*B*—O3*G*	27.4 (2)	C2*B*—O3*G*—C5—C4*B*	1.1 (3)
N1—C1*A*—C2*B*—C3*G*	30.5 (3)	C1*A*—C4*B*—C5—O3*G*	16.9 (2)

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D$$\cdots$$A$	D—H$\cdots$$A$
N1—H6$\cdots$O17^i	0.88	2.16	2.973 (3)	153

Symmetry code: (i) $x, 1 + y, z$.
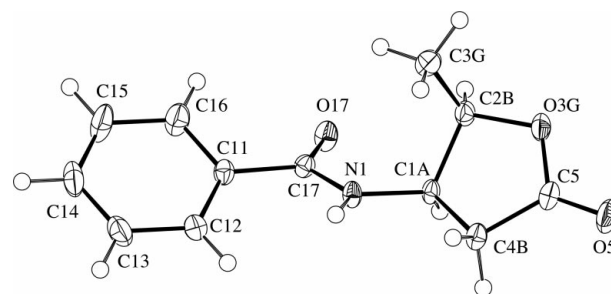
Figure 1
Structure of (I), with displacement ellipsoids drawn at the 50% probability level.
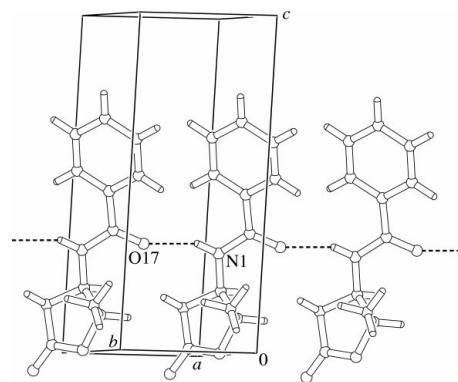
Figure 2
Packing diagram of (I). Dashed lines represent hydrogen bonds.

H atoms were placed at calculated positions (C—H = 0.95–1.00 Å and N—H = 0.88 Å), with isotropic displacement parameters [$U_{iso} = 1.5U_{eq}$(C) for methyl H atoms and $1.2U_{eq}$(parent atom) for all other atoms], and included in the structure-factor calculation. In the absence of significant anomalous scattering effects, Friedel pairs were merged, and the absolute configuration is assumed from that of the starting material.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PARST* (Nardelli, 1983).

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