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tert-Butoxycarbonyl-L-leucyl-L-threoninamide

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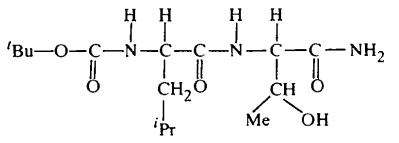
Abstract

The peptide chain in $C_{15}H_{29}N_3O_5$ adopts an extended conformation. The peptide unit is *trans* and shows significant deviations from planarity. The crystal packing enables neighbouring molecules to interact through hydrogen bonding in an anti-parallel fashion.

Comment

The conformation of the *tert*-butoxycarbonyl group in the title peptide, (I), is characterized by the torsion angles θ_0 = 175.6 (3)° (C1—O1—C0′—N1) and ω_0 = −173.7 (3)° (O1—C0′—N1—C1*A*) as *trans–trans* (Benedetti *et al.*, 1980). The peptide unit is *trans* and shows significant deviation from planarity ($\Delta\omega$ = 10.7°). The carboxyl group makes a dihedral angle of 37.5° with the adjacent peptide unit. The peptide chain backbone is folded with the conformation φ_1 = −119.8 (3), ψ_1 = 0.9 (4), ω_1 = 169.3 (3), φ_2 = −139.6 (3) and ψ_2 = 161.1 (3)°.



(I)

The side chain of the leucyl residue has torsion angles χ_1 = −58.9 (4), χ_{21} = 174.4 (3) and χ_{22} = −60.5 (5)°, corresponding to the commonly observed $g^-(tg^-)$ conformation (Benedetti *et al.*, 1983). The side-chain conformation of the threonyl residue is described by χ_{11} (N2—C2*A*—C2*B*—C2*G*) = −51.5 (3)° and χ_{12} (N2—C2*A*—C2*B*—O2*G*) = 73.2 (3)°. For comparison, in Gly–Thr–H_2O, χ_{11} = −62 and χ_{12} = 61° (Yadava & Padmanabhan, 1973) and in Boc–Phe–D-Leu–Thr–OMe, χ_{11} = −69.4 (5) and χ_{12} = 49.9 (4)° (Doi *et al.*, 1993).

The peptide chain folding introduces an intramolecular interaction [2.629 (3) Å] between N2 and O2′. Such an interaction and an accompanying large deviation from planarity of the peptide unit have been observed in other peptides, such as Leu–Leu (Mitra & Subramanian, 1994) and Leu–Ala (Mitra *et al.*, 1996).

The packing gives rise to layers of molecules perpendicular to the *c* axis, with three intermolecular N—H···O hydrogen bonds holding the molecules together in each layer, while the layers are stabilized through hydrophobic and van der Waals interactions.

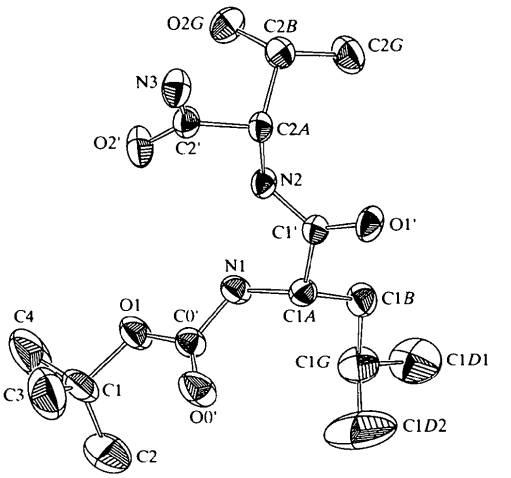


Fig. 1. Perspective view of the molecule, with displacement ellipsoids shown at the 50% probability level. H atoms have been omitted for clarity.

Experimental

The crystals were obtained by slow evaporation of a methanol/water solution at room temperature.

Crystal data

$C_{15}H_{29}N_3O_5$
M_r = 331.41
Orthorhombic
$P2_12_12_1$
a = 7.713 (2) Å
b = 8.674 (1) Å
c = 28.325 (6) Å
V = 1895.0 (7) Å^3
Z = 4
D_x = 1.162 Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 15–22°
μ = 0.719 mm^{-1}
T = 293 (2) K
Parallelepiped
0.40 × 0.28 × 0.20 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2140 measured reflections
2140 independent reflections
2030 reflections with $I > 2\sigma(I)$
$\theta_{max} = 73°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 35$
3 standard reflections
every 100 reflections
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.176$
$S = 1.008$
2137 reflections
215 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0708P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.357$ e Å^{-3}
$\Delta\rho_{min} = -0.249$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0040 (12)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = 0.1 (4)

Table 1. *Selected torsion angles (°)*

C1—O1—C0′—N1	175.6 (3)
O1—C0′—N1—C1*A*	−173.7 (3)
C0′—N1—C1*A*—C1′	−119.8 (3)
N1—C1*A*—C1*B*—C1*G*	−58.9 (4)
C1*A*—C1*B*—C1*G*—C1*D*2	−60.5 (6)
C1*A*—C1*B*—C1*G*—C1*D*1	174.4 (3)
N1—C1*A*—C1′—N2	0.9 (4)
C1*A*—C1′—N2—C2*A*	169.3 (3)
C1′—N2—C2*A*—C2′	−139.6 (3)
N2—C2*A*—C2*B*—O2*G*	73.2 (3)
N2—C2*A*—C2*B*—C2*G*	−51.5 (3)

Table 2. *Selected hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O2*G*^{i}	0.86	2.40	3.056 (3)	134
N3—H2N3···O1′^{ii}	0.86	2.17	2.941 (4)	163
O2*G*···O2′^{i}	–	–	2.750 (4)	–

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

All H-atom positions, except that of O2*G*, were geometrically fixed and allowed to ride on the corresponding non-H atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *SDP* (Frenz, 1978). Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1997). Software used to prepare material for publication: *SHELXL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: VJ1089). Services for accessing these data are described at the back of the journal.

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Dineopentylprehnitene (1,2,3,4-tetramethyl-5,6-dineopentylbenzene) at 296 and 223 K

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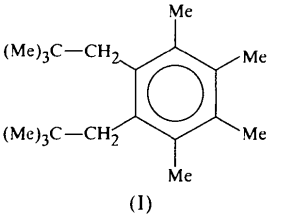
Abstract

The title substance, $C_{20}H_{34}$, crystallized in the centrosymmetric space group *C*2/*c* with a single molecule as the asymmetric unit. The molecule is quite crowded intramolecularly, and has a notably non-planar benzene ring. There is, however, virtually no intermolecular crowding.

Comment

Interest in dineopentylprehnitene, (I), is associated with its potential for substantial crowding in the crystalline state. It crystallized in the centrosymmetric space group



(I)