Molecule *B*				
C(1*b*)	0.5296 (4)	1.0365 (3)	0.7188 (4)	0.070 (3)
C(2*b*)	0.6143 (6)	1.0254 (4)	0.6371 (5)	0.100 (5)
C(3*b*)	0.4374 (5)	1.0972 (4)	0.6805 (5)	0.103 (5)
C(4*b*)	0.4597 (6)	0.9694 (4)	0.7321 (5)	0.112 (5)
O(5*b*)	0.6120 (3)	1.0473 (2)	0.8394 (3)	0.063 (2)
C(6*b*)	0.6809 (4)	1.1057 (3)	0.8664 (4)	0.056 (3)
O(7*b*)	0.6755 (3)	1.1587 (2)	0.8089 (3)	0.078 (2)
N(1)	0.7631 (3)	1.0971 (2)	0.9754 (3)	0.047 (2)
C(1*A*)	0.8344 (4)	1.1581 (2)	1.0321 (3)	0.047 (2)
C(1*B*)	0.8871 (4)	1.1410 (3)	1.1654 (4)	0.057 (3)
C(1*G*)	0.7913 (4)	1.1512 (3)	1.2353 (4)	0.059 (3)
C(1*D*1)	0.7341 (5)	1.0924 (4)	1.2726 (4)	0.081 (4)
C(1*E*1)	0.6478 (7)	1.0997 (5)	1.3394 (5)	0.103 (5)
C(1*Z*)	0.6223 (6)	1.1638 (5)	1.3768 (5)	0.104 (5)
C(1*E*2)	0.6708 (7)	1.2234 (5)	1.3407 (7)	0.122 (6)
C(1*D*2)	0.7617 (5)	1.2169 (3)	1.2685 (5)	0.084 (4)
C(1)	0.9476 (4)	1.1790 (2)	0.9841 (4)	0.047 (2)
O(1)	0.9926 (3)	1.2381 (2)	1.0101 (3)	0.065 (2)
N(2)	0.9889 (3)	1.1335 (2)	0.9175 (3)	0.052 (2)
C(2*A*)	1.1047 (4)	1.1462 (2)	0.8823 (4)	0.050 (3)
C(2*B*)	1.0992 (4)	1.1154 (3)	0.7602 (4)	0.060 (3)
C(2*G*)	0.9926 (4)	1.1448 (3)	0.6641 (4)	0.074 (3)
C(2*D*1)	0.9902 (7)	1.1039 (5)	0.5503 (5)	0.128 (6)
C(2*D*2)	0.9962 (7)	1.2225 (4)	0.6469 (6)	0.118 (6)
C(2)	1.2160 (5)	1.1172 (3)	0.9775 (4)	0.067 (3)
O(2)	1.2033 (4)	1.0865 (3)	1.0645 (4)	0.120 (4)
O(2*m*)	1.3224 (3)	1.1270 (3)	0.9554 (3)	0.114 (3)
C(2*m*)	1.4356 (5)	1.1045 (6)	1.0446 (5)	0.128 (6)

Table 2. *Selected torsion angles* (°) *with e.s.d.'s in parentheses*

		Molecule *A*	Molecule *B*
Phe	φ*	−80.4 (4)	−77.2 (4)
	ψ	−55.6 (4)	−14.9 (4)
	ω	−173.5 (5)	−171.9 (4)
	χ^1	−59.4 (4)	−80.8 (4)
	χ^2	−68.0 (5)	−78.2 (5)
Leu	φ	72.8 (5)	87.4 (4)
	ψ†	17.3 (7)	2.5 (5)
	ω‡	−174.0 (8)	176.7 (4)
	χ^1	51.6 (6)	58.7 (4)
	χ^2	−178.1 (8)	−174.1 (5)

* C(6*b*)—N(1)—C(1*A*)—C(1).
† N(2)—C(2*A*)—C(2)—O(2).
‡ C(2*A*)—C(2)—O(2*m*)—C(2*m*).

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986). During the last stage of refinement all H atoms were placed at assumed positions (C—H = 1.08 and N—H = 0.97 Å, $U = 1.2 \times U_{eq}$ of associated non-H atoms) and refined. Refinement was by full-matrix least-squares methods using *SHELX*76 (Sheldrick, 1976). The *y* coordinate of atom C(1*b*) of molecule *A* was fixed during the refinement. The molecular conformation was drawn by the *ORTEP* program (Johnson, 1971).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond distances and angles, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71109 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1034]

References

Johnson, C. K. (1971). *ORTEP*II. Report ORNL-3974, revised. Oak Ridge National Laboratory, Tennessee, USA.

Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1986). *SHELXS*86. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

Zajac, J. M., Gacel, G., Petit, F., Dodey, P., Rossignol, P. & Roques, B. P. (1983). *Biochem. Biophys. Res. Commun.* **111**, 390–397.

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Structure of Boc-Phe-D-Leu-Thr-OMe

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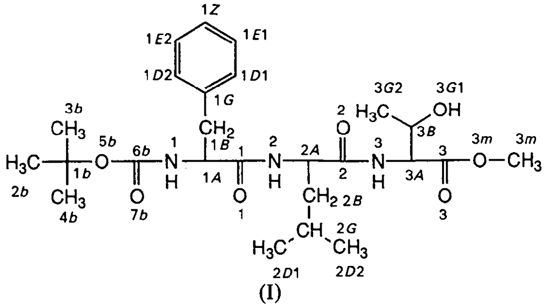
Abstract

The X-ray crystal structure analysis shows that *tert*-butoxycarbonyl-L-phenylalanyl-D-leucine-L-threonine methyl ester takes an open conformation in which the *tert*-butoxycarbonyl group is located face-to-face with the D-leucine isobutyl side chain. In the crystal, the molecules, translated by twofold screw symmetry, form an infinite sheet structure through four independent hydrogen bonds.

Comment

As one of a series of investigations into the relationship between the molecular conformation and the hydrophobicity/hydrophilicity of oligopeptides consisting of L-L, L-D or D-L sequences, the title peptide (I) was chemically synthesized and its molecular conformation determined by X-ray crystal analysis. The conformational study is also interesting in determining whether or not the conformational characteristic observed in the related Boc-Phe-D-Leu-OMe peptide (Doi, In, Ikuma, Inoue & Ishida, 1993), *i.e.* the face-to-face alignment between the *tert*-butoxycarbonyl and D-leucine isobutyl side groups, is maintained.



(I)

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The atomic positional and equivalent isotropic thermal parameters are listed in Table 1. Table 2 lists some conformational torsion angles. Fig. 1 shows a stereo diagram of the molecular conformation. The Boc-Phe-D-Leu-Thr-OMe molecule takes an open conformation in such a way that the N-terminal *tert*-butoxycarbonyl group is arranged side by side with the D-Leu isobutyl side chain and is orientated anitparallel to the Thr-OMe backbone chain. Respective φ and ψ angles of Phe, D-Leu and Thr residues are all in the allowed regions (Ramachandran, Ramakrishnan & Sasisekharan, 1963). It is noteable that the prominent overlapping between the *tert*-butoxycarbonyl and Leu isobutyl groups, which is characteristic of the molecular conformation of Boc-Phe-D-Leu-OMe, is also formed without any significant effect of a hydrophilic Thr residue, and this would reflect the intrinsic conformational feature of the Boc-Phe-D-Leu sequence. Although the bonding parameters have relatively high e.s.d.'s because of the large thermal motion, especially for the terminal chains (0.005–0.01 Å for the bond lengths and 0.2–0.7° for the bond angles), their values are all in the accepted regions, and no notable abnormality was observed.

In the crystal structure, the molecules translated by twofold screw symmetry form an infinite anti-parallel sheet structure through four hydrogen bonds, as is shown in Fig. 2, *i.e.* N(1)···O(2) (at $-x$, $y-\frac{1}{2}$, $\frac{3}{2}-z$) = 2.867 (5); N(2)···O(1)(at $-x$, $y-\frac{1}{2}$, $\frac{3}{2}-z$) = 3.002 (5); N(3)···O(1)(at $-x$, $y-\frac{1}{2}$, $\frac{3}{2}-z$) = 3.043 (5); O(3*G*1)···O(7*b*)(at $-x$, $y-\frac{1}{2}$, $\frac{3}{2}-z$) = 2.747 (6) Å. Although the peptide was cocrystallized with DMF (dimethylformamide) solvent, no specific interaction was observed; the solvent stabilized the crystal structure through short contacts with the C-terminal OMe moiety.

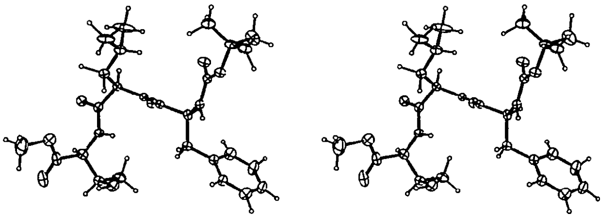

Fig. 1. Stereoscopic view of the Boc-Phe-D-Leu-Thr-OMe molecule.

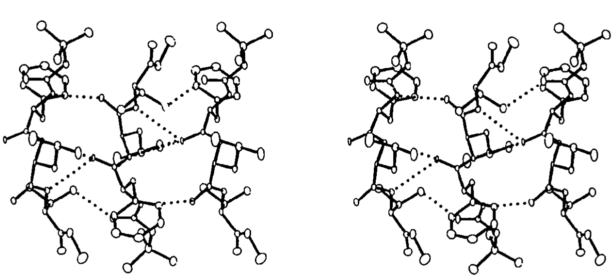

Fig. 2. Stereoscopic view of the molecular arrangement of Boc-Phe-D-Leu-Thr-OMe translated by twofold screw symmetry. The dotted lines represent hydrogen bonds.

Experimental

Crystal data

$C_{28}H_{46}N_4O_8$
M_r = 566.69
Orthorhombic
$P2_12_12_1$
a = 29.807 (10) Å
b = 9.469 (2) Å
c = 11.515 (2) Å
V = 3250 (1) Å^3
Z = 4
D_x = 1.158 Mg m^{-3}
D_m = 1.150 (5) Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 22–26°
μ = 0.663 mm^{-1}
T = 293 K
Plates
0.3 × 0.15 × 0.1 mm
Transparent

Data collection

Rigaku AFC-5 diffractometer
ω-2θ scans
Absorption correction: none
3654 measured reflections
3155 independent reflections
2333 observed reflections [$I > 2\sigma(I)$]

R_{int} = 0.018
$\theta_{\max}$ = 64.9°
$h = 0 \rightarrow 34$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 13$
4 standard reflections monitored every 100 reflections
intensity variation: 4%

Refinement

Refinement on F
Final R = 0.058
wR = 0.065
S = 0.804
2333 reflections
361 parameters
H-atom parameters not refined

$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{\max}$ = 0.41
$\Delta\rho_{\max}$ = 0.28 e Å^{-3}
$\Delta\rho_{\min}$ = −0.22 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\text{eq}} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1*b*)	0.8749 (3)	0.6224 (6)	0.6562 (5)	0.066 (4)
C(2*b*)	0.8391 (4)	0.5016 (9)	0.6696 (9)	0.110 (7)
C(3*b*)	0.8554 (3)	0.7647 (8)	0.6853 (6)	0.081 (5)
C(4*b*)	0.9171 (3)	0.5874 (7)	0.7235 (6)	0.080 (5)
O(5*b*)	0.8861 (1)	0.6385 (4)	0.5320 (3)	0.057 (2)
C(6*b*)	0.9034 (2)	0.5327 (5)	0.4686 (4)	0.042 (3)
O(7*b*)	0.9103 (2)	0.4123 (4)	0.5068 (4)	0.065 (2)
N(1)	0.9094 (1)	0.5668 (4)	0.3588 (3)	0.038 (2)
C(1*A*)	0.9240 (2)	0.4609 (5)	0.2749 (5)	0.043 (3)
C(1*B*)	0.9176 (2)	0.5118 (5)	0.1492 (4)	0.046 (3)
C(1*G*)	0.8704 (2)	0.5202 (6)	0.1083 (5)	0.050 (3)
C(1*D*1)	0.8523 (2)	0.6432 (6)	0.0593 (6)	0.058 (3)
C(1*E*1)	0.8113 (3)	0.6481 (7)	0.0162 (7)	0.078 (4)
C(1*Z*)	0.7829 (2)	0.5286 (9)	0.0199 (7)	0.087 (5)
C(1*E*2)	0.8000 (2)	0.4064 (9)	0.0656 (9)	0.099 (6)
C(1*D*2)	0.8414 (2)	0.4009 (6)	0.1104 (7)	0.072 (4)
C(1)	0.9723 (2)	0.4144 (5)	0.2892 (4)	0.036 (2)
O(1)	0.9841 (1)	0.2972 (3)	0.2575 (3)	0.046 (2)

N(2)	1.0013 (1)	0.5056 (3)	0.3375 (3)	0.032 (2)
C(2*A*)	1.0474 (2)	0.4652 (4)	0.3553 (4)	0.034 (2)
C(2*B*)	1.0725 (2)	0.5727 (5)	0.4335 (4)	0.048 (3)
C(2*G*)	1.0524 (2)	0.5866 (6)	0.5511 (5)	0.062 (4)
C(2*D*1)	1.0793 (3)	0.6983 (6)	0.6199 (5)	0.089 (5)
C(2*D*2)	1.0504 (4)	0.4506 (9)	0.6163 (6)	0.136 (7)
C(2)	1.0726 (2)	0.4441 (4)	0.2440 (5)	0.043 (3)
O(2)	1.1008 (1)	0.3464 (3)	0.2331 (3)	0.053 (2)
N(3)	1.0658 (1)	0.5366 (4)	0.1573 (3)	0.042 (2)
C(3*A*)	1.0870 (2)	0.5264 (5)	0.0470 (5)	0.051 (3)
C(3*B*)	1.0538 (2)	0.5597 (5)	−0.0495 (4)	0.051 (3)
O(3*G*1)	1.0312 (1)	0.6890 (4)	−0.0235 (4)	0.071 (3)
C(3*G*2)	1.0188 (2)	0.4404 (7)	−0.0582 (5)	0.070 (4)
C(3)	1.1295 (2)	0.6165 (5)	0.0367 (7)	0.066 (4)
O(3)	1.1469 (1)	0.6221 (5)	−0.0595 (5)	0.100 (4)
O(3*m*)	1.1457 (2)	0.6636 (4)	0.1307 (5)	0.077 (3)
C(3*m*)	1.1899 (3)	0.736 (1)	0.106 (1)	0.150 (8)
C(1*df*)	0.7891 (4)	0.9673 (9)	0.198 (1)	0.126 (8)
O(2*df*)	0.7727 (3)	1.0450 (8)	0.1243 (8)	0.195 (8)
N(3*df*)	0.7649 (2)	0.8986 (7)	0.2784 (7)	0.104 (5)
C(4*df*)	0.7171 (3)	0.911 (2)	0.300 (1)	0.18 (1)
C(5*df*)	0.7880 (3)	0.805 (1)	0.354 (1)	0.142 (8)

Table 2. *Selected torsion angles (°) with e.s.d.'s in parentheses*

	Phe	D-Leu	Thr
φ	−71.0 (4)*	66.8 (4)	−95.6 (5)
ψ	−26.8 (4)	41.4 (4)	14.3 (6)†
ω	178.7 (4)	−178.8 (5)	174.0 (6)‡
χ^1	−71.2 (4)	60.3 (4)	49.9 (4)¶
χ^2	−57.0 (6)	57.9 (5)	

* C(6*b*)—N(1)—C(1*A*)—C(1).
† N(3)—C(3*A*)—C(3)—O(3*m*).
‡ C(2*A*)—C(2)—O(2*m*)—C(2*m*).
¶N(3)—C(3*A*)—C(3*B*)—O(3*G*1).

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1986). During the last stage of refinement all H atoms were placed at assumed positions with an overall isotropic thermal parameter (U = 0.05 Å^2) and were only included for structure-factor calculations. Refinement of non-H atoms was by full-matrix least-squares methods using *SHELX*76 (Sheldrick, 1976). The molecular conformation was drawn by the *ORTEP* program (Johnson, 1971).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond distances and angles, and torsion angles have been deposited with the british Library Document Supply Centre as Supplementary Publication No. SUP 71108 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1046]

References

Doi, M., In, Y., Ikuma, K., Inoue, M. & Ishida, T. (1993). *Acta Cryst.* C**49**, 1528–1530.
Johnson, C. K. (1971). *ORTEP*II. Report ORNL-3974. Revised. Oak Ridge National Laboratory, Tennessee, USA.
Ramachandran, G. N., Ramakrishnan, C. & Sasisekharan, V. (1963). *J. Mol. Biol.* 7, 95–99.
Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
Sheldrick, G. M. (1986). *SHELXS*86. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

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Structure of 2-(1-Naphthyl)-5-phenyl-1,3-oxazole (α-NPO)

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Abstract

The α-NPO molecule is planar with an average deviation of non-H atoms from the least-squares plane of 0.02 (2) Å.

Comment

The laser output and lifetime of the title compound has been studied and successfully improved (Konjevic, 1988). Since the structure of the dye plays a significant role in its photophysical properties, the structure of α-NPO was determined as part of our structural laser-dye studies. The naphthyl, oxazole and phenyl rings of the molecule are coplanar with an average deviation of non-H atoms from the least-squares plane of 0.02 (2) Å. The bond lengths and angles in the oxazole ring of this compound are comparable with those of 2-amino-1,3-oxazole (Albinati & Marcon, 1981). The C—C bonds and C—C—C angles within the phenyl and naphthyl rings are typical of such systems, ranging from

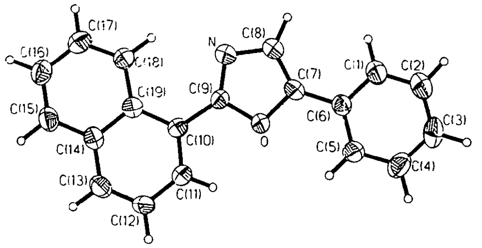

Fig. 1. A view of the molecule showing the labeling of the non-H atoms. Thermal ellipsoids are shown at the 50% probability level.

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