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The Structure of *tert*-Butoxycarbonyl-L-prolyl-L-alanylglycinamide

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Abstract

The crystal structure of the title compound, $C_{15}H_{26}N_4O_5$, $M_r = 342{\cdot}45$, has been determined by the X-ray method. The space group is $P2_12_12_1$, with $a = 10{\cdot}078$ (1), $b = 19{\cdot}246$ (1), $c = 9{\cdot}272$ (1) Å, $Z = 4$, $D_m = 1{\cdot}25$, $D_c = 1{\cdot}27$ Mg m^{-3}, m.p. = 494–495 K. The final R value was 0·056 for 2009 reflections with $2\theta \leq 150°$. The main chain of this peptide is folded at the Ala-Gly site into the β-turn type (I), in contrast to an extended form of the similar sequential peptide pentyloxycarbonyl(Poc)-Pro-Ala-Gly-OH. The structure of this peptide has revealed that the widening of the $NC^{\alpha}C'$ angle of the second residue of the β-turn type (I) is highly significant for any kind of residue.

Introduction

X-ray studies have shown that the linear oligopeptides having the -Pro-*X*-Gly- sequence (*X*: any amino acid residue) usually take one of the three conformations β-sheet, β-turn and polyproline (II)-type helix. *tert*-Butoxycarbonyl(Boc)-Pro-Pro-Gly-OH (Hudson, Shaw, Schurr & Jensen, 1972) has a polyproline (II)-type helix. Several peptides having the -Pro-Leu-Gly- sequence (Ashida, Tanaka, Shimonishi & Kakudo, 1977) and Boc-Pro-Pro-Gly-NH_2 (Tanaka, Ashida, Shimonishi & Kakudo, 1979) are folded into the typical β-turn of type (I). Boc-Pro-Ile-Gly-OH (Yamada, Tanaka & Ashida, 1980) and Boc-Pro-Val-Gly-OH (Tanaka & Ashida, 1980), both with a branched side chain at the C^{β} atom, and Poc-Pro-Ala-Gly-OH (Yamada, Tanaka & Ashida, 1981) have the extended conformation of the antiparallel β-sheet type. However, the last one, Poc-Pro-Ala-Gly-OH, may sometimes take a β-turn conformation, since the alanyl residue has only a single methyl group as a side chain.

In this study, therefore, Boc-Pro-Ala-Gly-NH_2, modified at the C-terminal end of Boc-Pro-Ala-Gly-OH, was prepared and its structure was investigated by the

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X-ray method. If it takes the β-turn, two conformations are possible; one has an intramolecular hydrogen bond between NH of Gly and CO of Boc, and the other has a bond between the C-terminal NH and CO of Pro.

Experimental

tert-Butoxycarbonyl-L-prolyl-L-alanylglycinamide was prepared by the amination of the *p*-nitrophenyl ester previously obtained by the dehydration reaction of *tert*-butoxycarbonyl-L-prolyl-L-alanylglycine with *p*-nitrophenol, and crystallized from an ethanol/water solution.

A crystal of dimensions 0·4 × 0·3 × 0·2 mm was used in the experiment. The lattice parameters were determined by a least-squares refinement from 2θ values of high-angle reflections. The intensity data were collected on a Rigaku four-circle diffractometer in the Ultra High Intensity X-ray Laboratory of this university. The θ–2θ continuous-scan method was adopted with graphite-monochromated Cu $K\alpha$ radiation. The symmetrical A setting for the reflections with $0 < 2\theta < 132°$ and fixed χ setting (Arndt & Willis, 1966) for those with $132 < 2\theta < 150°$ were adopted. Of all 2009 reflections up to 150° (2θ) excluding some reflections in the blind regions of the device, 1984 were non-zero. Lorentz and polarization factors were applied, but no absorption corrections were made.

Structure determination

The structure was solved by the direct method with the program *MULTAN* 78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). On the E map calculated from the best set of the phase angles, 22 of the 24 non-hydrogen atoms were located. A difference Fourier synthesis revealed the two remaining C atoms. The refinement was carried out by the block-diagonal least-squares method with *HBLS* V (Ashida, 1973). All the H atoms were obtained on a ΔF map, and were included in the refinement.

The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The final R value was 0·056 for all reflections, and 0·055 for the non-zero reflections. The function minimized was $\sum w(|F_o| - |F_c|)^2$, with $w = 0{\cdot}4411$ for $|F_o| = 0$ and $w = [\sigma^2(F) + 0{\cdot}0378|F_o| + 0{\cdot}0007|F_o|^2]^{-1}$ for $|F_o| > 0$, where $\sigma(F)$ is the standard deviation based on the counting statistics. 31 reflections were corrected for the secondary-extinction effect (secondary-extinction coefficient, $g = 0{\cdot}462 \times 10^{-5}$, Zachariasen, 1967). All the calculations were made on the FACOM M-200 computer of this university. The final positional parameters are listed in Tables 1 and 2.*

* Lists of structure factors, anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36289 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic parameters* ($\times 10^4$, $B_{eq} \times 10$) *with their e.s.d.'s in parentheses for the non-*H *atoms*

	x	*y*	*z*	B_{eq} (Å²)
C(1)	9510 (7)	1072 (3)	3045 (6)	68 (3)
C(2)	7728 (5)	399 (5)	1742 (7)	82 (4)
C(3)	9420 (6)	−225 (3)	3193 (5)	66 (3)
C(4)	9146 (5)	412 (3)	2274 (4)	48 (2)
O(1)	10062 (3)	425 (1)	1040 (2)	39 (1)
C(5)	10067 (4)	−68 (2)	23 (4)	36 (1)
O(2)	9544 (4)	−640 (2)	99 (4)	59 (2)
N(1)	10789 (3)	138 (1)	−1107 (3)	34 (1)
C(6)	11230 (3)	854 (1)	−1294 (3)	30 (1)
C(7)	11869 (5)	845 (2)	−2801 (4)	49 (2)
C(8)	11414 (10)	218 (4)	−3479 (6)	98 (4)
C(9)	10960 (6)	−286 (2)	−2402 (4)	49 (2)
C(10)	10061 (3)	1367 (1)	−1229 (3)	27 (1)
O(3)	8969 (2)	1218 (1)	−1704 (3)	36 (1)
N(2)	10367 (3)	1988 (1)	−678 (3)	28 (1)
C(11)	9334 (3)	2493 (2)	−351 (3)	32 (1)
C(12)	9940 (6)	3108 (3)	427 (7)	69 (3)
C(13)	8566 (3)	2743 (1)	−1657 (3)	27 (1)
O(4)	7440 (2)	2992 (1)	−1474 (3)	39 (1)
N(3)	9116 (2)	2703 (1)	−2952 (3)	27 (1)
C(14)	8394 (3)	2903 (2)	−4237 (3)	32 (1)
C(15)	7355 (3)	2391 (2)	−4758 (3)	34 (1)
O(5)	6668 (4)	2559 (2)	−5778 (4)	60 (2)
N(4)	7285 (3)	1777 (1)	−4111 (3)	36 (1)

Table 2. H-*atom positional parameters* ($\times 10^3$) *with their e.s.d.'s in parentheses*

	x	*y*	*z*	Bonded to
H(1)	889 (8)	114 (4)	389 (9)	C(1)
H(2)	924 (7)	148 (4)	242 (8)	C(1)
H(3)	1049 (8)	106 (4)	332 (9)	C(1)
H(4)	715 (9)	42 (5)	269 (8)	C(2)
H(5)	749 (13)	−6 (6)	113 (9)	C(2)
H(6)	759 (9)	84 (4)	119 (9)	C(2)
H(7)	885 (8)	−21 (4)	396 (8)	C(3)
H(8)	1032 (7)	−18 (4)	359 (8)	C(3)
H(9)	926 (9)	−72 (4)	257 (9)	C(3)
H(10)	1186 (5)	99 (2)	−51 (5)	C(6)
H(11)	1291 (7)	82 (3)	−278 (7)	C(7)
H(12)	1161 (6)	129 (3)	−334 (8)	C(7)
H(13)	1217 (9)	−4 (5)	−409 (9)	C(8)
H(14)	1041 (9)	36 (5)	−413 (10)	C(8)
H(15)	1175 (7)	−65 (3)	−225 (7)	C(9)
H(16)	1010 (6)	−53 (3)	−268 (8)	C(9)
H(17)	1110 (6)	202 (3)	3 (6)	N(2)
H(18)	869 (6)	223 (3)	27 (6)	C(11)
H(19)	919 (8)	346 (4)	61 (8)	C(12)
H(20)	1079 (8)	332 (4)	−32 (8)	C(12)
H(21)	1045 (8)	296 (4)	141 (9)	C(12)
H(22)	1001 (6)	257 (3)	−325 (6)	N(3)
H(23)	905 (6)	296 (3)	−506 (6)	C(14)
H(24)	796 (5)	337 (3)	−405 (6)	C(14)
H(25)	793 (6)	164 (3)	−320 (7)	N(4)
H(26)	663 (5)	145 (3)	−442 (6)	N(4)

Discussion

The crystal structure projected along **c** is shown in Fig. 1. The bond lengths, angles and torsion angles are given in Fig. 2. The hydrogen bonds are listed in Table 3.

Most of the bond lengths and angles are in good agreement with those of the similar oligopeptides. The C^{γ} atom of Pro has an unusually large thermal vibration perpendicular to the pyrrolidine ring plane. The atom may have a kind of disordered structure, which may explain the very short C^{β}–C^{γ} and C^{γ}–C^{δ} distances and very wide C^{β}–C^{γ}–C^{δ} angle. Such an example has been reported elsewhere (Ashida & Kakudo, 1974).

As is shown in Fig. 2, the main chain of this peptide is folded at the Ala-Gly site into the β-turn type (I), which contrasts remarkably with an extended conformation of the similar sequential peptide Poc-Pro-Ala-Gly-OH (Yamada *et al.*, 1981). The overall shapes of this peptide possess a very similar conformation to the folded one of Boc-Pro-Pro-Gly-NH_2 (Tanaka *et al.*, 1979): a polyproline (II)-type structure at the N-terminal half of the molecule and a β-turn structure at the C-terminal half with the amide group as a hydrogen donor. These two molecules are compared in Fig. 3. A more precise comparison showed only a little difference in the torsion angles of C'–$N(\omega_{Boc\text{-}Pro})$ and C^{α}–$C'(\psi_{Gly})$; the corresponding values of Boc-Pro-Pro-Gly-NH_2 are $\omega_{Boc\text{-}Pro} = -2{\cdot}2$, $\psi_{Gly} = 6{\cdot}1°$.

The torsion angles (φ, ψ) of the second and third residues in the β-turn structures of the linear oligopeptides are listed in Table 4 and are plotted on the (φ, ψ) map in Fig. 4. In the linear oligopeptides it is rare that the second position of the β-turn type (I) is occupied by the non-Pro residue. However, as is shown in Table 4, (φ_2, ψ_2) values for Ala are almost equal to those for Pro.

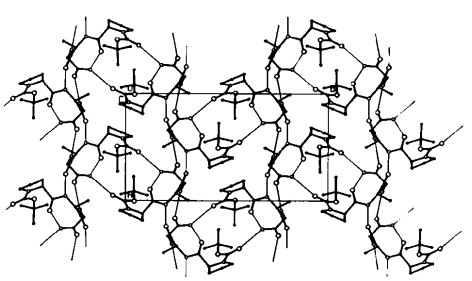

Fig. 1. The crystal structure of Boc-Pro-Ala-Gly-NH_2 viewed along **c**. The hydrogen bonds are shown by thin lines.

Table 3. *Hydrogen bonds*

D–H···*A*	*D*···*A*	H···*A*	*D*–H···*A*
N(2)–H(17)···O(4^{i})	2·889 (4) Å	1·90 (6) Å	175 (5)°
N(3)–H(22)···O(5^{ii})	2·873 (4)	1·92 (6)	167 (5)
N(4)–H(25)···O(2^{iii})	2·953 (5)	2·01 (6)	170 (5)
N(4)–H(26)···O(3)	3·003 (4)	1·92 (6)	169 (5)

Symmetry code: (i) 0·5 + *x*, 0·5 − *y*, −*z*; (ii) 0·5 + *x*, 0·5 − *y*, 1·0 − *z*; (iii) 1·5 − *x*, −*y*, −0·5 + *z*.

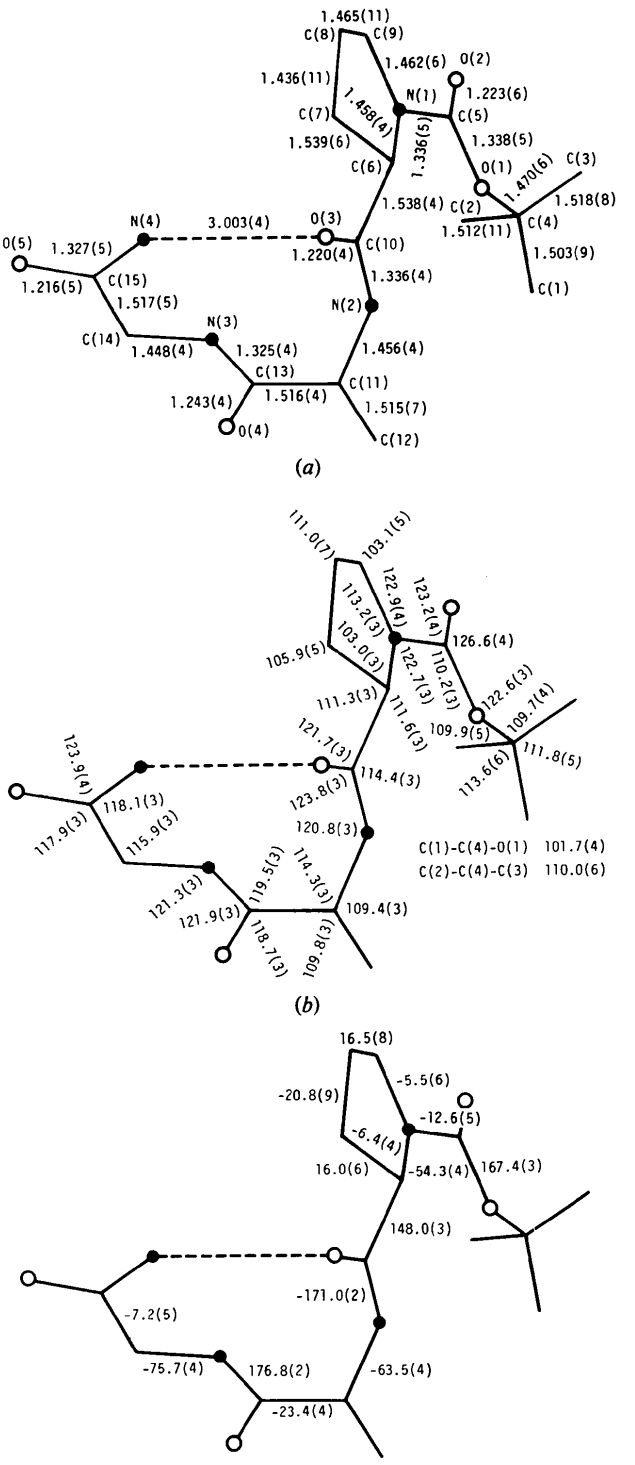

Fig. 2. (*a*) Bond lengths (Å), (*b*) bond angles (°), and (*c*) torsion angles (°).

Table 4. *Main-chain torsion angles in the β-turn* (°)

R_1 R_2 R_3 R_4	φ_2	ψ_2	φ_3	ψ_3	Reference
Type (I)					
Boc-Pro-Ala-Gly-NH_2	−63·5 (4)	−23·4 (4)	−75·7 (4)	−7·2 (5)	This study
Boc-Pro-Pro-Gly-NH_2	−64·9	−23·0	−88·8	6·1	Tanaka *et al.* (1979)
Boc-Pro-Leu-Gly-OH	−65·0	−20·7	−110·8	26·7	Ashida *et al.* (1977)
S-Bzl-Cys-Pro-Leu-Gly-NH_2(*A*)*	−70·3	−16·0	−74·1	−8·5	Rudko & Low (1975)
S-Bzl-Cys-Pro-Leu-Gly-NH_2(*B*)*	−63·9	−27·9	−71·6	−11·9	Rudko & Low (1975)
S-Bzl-Cys-Pro-Leu-Gly-NH_2(*B*)*	−71·6	−11·9	−75·8	−16·2	Rudko & Low (1975)
(*p*-Br)Z-Gly-Pro-Leu-Gly-OH	−57·7	−33·4	−104·0	8·0	Ueki, Ashida, Kakudo, Sasada & Katsube (1969)
(*o*-Br)Z-Gly-Pro-Leu-Gly-Pro-OH	−65·0	−26·7	−104·7	8·4	Ueki, Bando, Ashida & Kakudo (1971)
Z-Gly-Pro-Leu-Gly-Pro-OH	−63·2	−23·0	−107·3	12·0	Bando, Tanaka, Ashida & Kakudo (1978)
Type (II)					
H-Pro-Leu-Gly-NH_2	−61·2	127·8	71·8	–	Reed & Johnson (1973)
Boc-Val-Pro-Gly-Gly-OBzl	−62·3	135·5	75·4	2·8	Ayato, Tanaka & Ashida (1981)

* Some of the torsion angles for these molecules were incorrectly given in the original paper. The angles recalculated from the published atomic coordinates are given here.

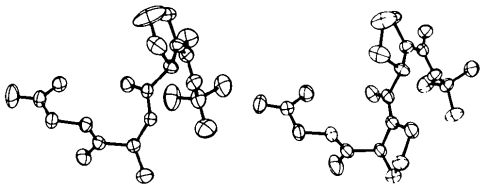

Fig. 3. A comparison of Boc-Pro-Ala-Gly-NH_2 (left) and Boc-Pro-Pro-Gly-NH_2 (right). The thermal ellipsoids are scaled to include 50% probability.

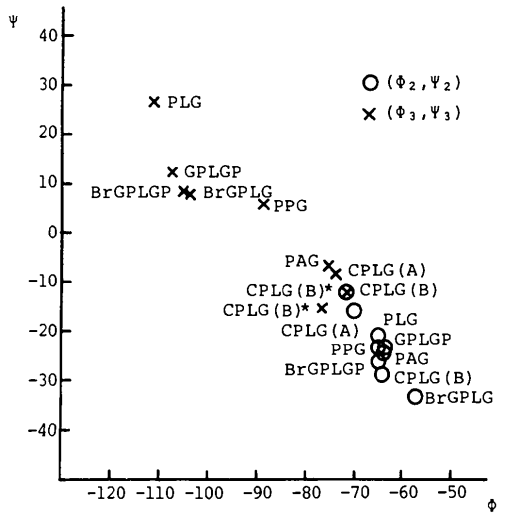

Fig. 4. Ramachandran plot of the main-chain torsion angles of the β-turn type (I). Open circles refer to the angles (φ_2,ψ_2), the torsion angles for the second residue, and crosses refer to the angles (φ_3,ψ_3), the torsion angles for the third residue. PAG (Boc-Pro-Ala-Gly-NH_2), PPG (Boc-Pro-Pro-Gly-NH_2), PLG (Boc-Pro-Leu-Gly-OH), GPLGP (Z-Gly-Pro-Leu-Gly-Pro-OH), BrGPLGP [(*o*-Br)Z-Gly-Pro-Leu-Gly-Pro-OH], BrGPLG [(*p*-Br)Z-Gly-Pro-Leu-Gly-OH], CPLG(*A*) and CPLG(*B*) (*S*-benzyl-Cys-Pro-Leu-Gly-NH_2 *A* and *B* molecules). CPLG(*B*)* is for Leu and Gly of a second β-turn.

The $NC^{\alpha}C'$ angle of the second residue, 114·3° for Ala, is greater than that of Poc-Pro-Ala-Gly-OH (Yamada *et al.*, 1981), 109·1°; the former is in accordance with the mean angles 113·6° for non-Pro residues at the second site of the β-turn type (I) in the cyclic oligopeptides, and 115·1° for the Pro residues (Ashida *et al.*, 1977). Thus it is clear that the widening of the $NC^{\alpha}C'$ angle of the second residue of the β-turn type (I) is highly significant for any kind of residue.

In most oligopeptides with three or more amino acid residues having a C-terminal amide group, such as *S*-benzyl-Cys-Pro-Leu-Gly-NH_2 (Rudko & Low, 1975), H-Pro-Leu-Gly-NH_2 (Reed & Johnson, 1973), isobutyl-Pro-Ala-isopropylamide (Aubry, Protas, Boussard & Marraud, 1977), pivaloyl-D-Pro-L-Pro-L-Ala-*N*-methylamide (Nair, Vijayan, Venkatachalapathy & Balaram, 1979) and Boc-Pro-Pro-Gly-NH_2 (Tanaka *et al.*, 1979), the C-terminal amide group commonly takes part in making a β-turn, except for *S*-benzyl-Cys-Pro-Leu-Gly-NH_2 (*A*). This suggests that the β-turn is one of the favorite conformations for peptides having aminated C-terminals.

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Etude Structurale par Diffraction des Rayons X du Désoxy-7 D-*glycéro*-β-D-*galacto*-heptopyrannoside de Méthyle Monohydraté et du Désoxy-7 L-*glycéro*-β-D-*galacto*-heptopyrannoside de Méthyle

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Abstract

Crystals of methyl 7-deoxy-D-*glycero*-β-D-*galacto*-heptopyranoside monohydrate, $C_8H_{16}O_6.H_2O$ (I), and methyl 7-deoxy-L-*glycero*-β-D-*galacto*-heptopyranoside, $C_8H_{16}O_6$ (II), are, respectively, monoclinic, space group $C2$, and orthorhombic, space group $P2_12_12_1$, with $a = 15{\cdot}63$ (1), $b = 4{\cdot}711$ (4), $c = 16{\cdot}24$ (3) Å, $\beta = 109{\cdot}09$ (12)°, $Z = 4$, $d_c = 1{\cdot}33$ Mg m^{-3} for (I) and $a = 25{\cdot}05$ (2), $b = 7{\cdot}758$ (6), $c = 5{\cdot}083$ (4) Å, $Z = 4$, $d_c = 1{\cdot}40$ Mg m^{-3} for (II). Both structures have been determined by direct methods and refined by least-squares calculations with Mo $K\alpha$ data to final R values of 0·038 (1101 reflexions) for (I) and 0·077 (861 reflexions) for (II). The hydroxyl group O(6)H and the methyl group C(7)H$_3$ are in the *gauche–trans* and *trans–gauche* conformations, respectively, relative to O(4) and O(5), for compound (I), in the reverse positions for compound (II). In both crystals, molecules are associated by hydrogen bonds in 'hydrophilic' regions clearly separated from 'hydrophobic' zones.

Introduction

Les composés (I) et (II) dont nous avons entrepris les études structurales et conformationnelles sont des produits de synthèse.

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