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Structure of an α,β-Unsaturated Dipeptide, Racemic *N*-[(Phenylmethoxy)carbonyl]-phenylalanyl-Δ^2-phenylalanine

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Abstract. $C_{26}H_{24}N_2O_5$, $M_r = 444{\cdot}49$, m.p. 466–469 K, triclinic, $P\bar{1}$, $a = 6{\cdot}418$ (1), $b = 13{\cdot}223$ (2), $c = 14{\cdot}153$ (2) Å, $\alpha = 104{\cdot}73$ (1), $\beta = 94{\cdot}33$ (2), $\gamma = 96{\cdot}54$ (2)°, $V = 1147{\cdot}2$ (3) Å^3, $Z = 2$, $D_x = 1{\cdot}287$ Mg m^{-3}, λ(Mo $K\bar{\alpha}$) = 0·71069 Å, $\mu = 0{\cdot}01$ mm^{-1}, $F(000) = 468$. Final $R = 0{\cdot}039$ for 2806 observed [$I \geq 3\sigma(I)$] reflections measured on a diffractometer at 293 K. The molecules in the crystal form compact 'hydrophilic columns' consisting of intermolecular hydrogen-bonded double chains surrounded by a hydrophobic space composed of phenyl substituents. The conformation of the phenylalanyl residue is close to parallel β-sheet ($\varphi = -89{\cdot}3$, $\psi = 114{\cdot}4$°) while the unsaturated residue adopts a left-handed α-helix conformation ($\varphi = 50{\cdot}4$, $\psi = 21{\cdot}2$°).

Introduction. α,β-Unsaturated amino acids have been found in many biologically active peptides, most of them having antibiotic properties (Noda, Shimohigashi & Izumiya, 1983). These dehydro amino acids are intermediates in the interchange of natural L-amino acids to their D enantiomers, as D molecules cannot be incorporated directly into the peptide chain (Demain, 1966). That is why a great number of microbial peptides having antibiotic activity contain both α,β-unsaturated and D-amino acid residues. Incorporation of a dehydro amino acid unit into the peptide decreases conformational flexibility and has a stabilizing influence on a β-turn (Aubry, Boussard & Marraud, 1984). The phenomenon has been used in analogues of biologically active peptides to reduce their enzymatic degradation (English & Stammer, 1978).

The structures of several dehydro amino acids [Z-Δ^E-Phe-OEt (Nitz, Holt, Rubin & Stammer, 1981); *N*-acetyl-Δ^Z-Phe-methylamide (Aubry, Allier, Boussard & Marraud, 1985); *N*-Boc-Δ^Z-Leu-OH (Chauhan, Stammer, Norskov-Lauritzen & Newton, 1979); *N*-acetyl-Δ^Z-Phe-OH (Ajò, Cesarin, Granozzi & Busetti, 1981); *N*-acetyl-Δ^Z-Phe-OEt (Ajò, Busetti, Ottenheijm & Plate, 1984); *N*-acetyl-Δ^Z-Pro-OH (Ajò, Busetti, Granozzi & Liakopoulou-Kyriakides, 1984); *N*-acetyl-Δ-Ala-OH (Ajò, Granozzi, Tondello, Del Pra & Zanotti, 1979)] and di- or tripeptides [*N*-acetyl-Δ^Z-Phe-Pro-OH (Ajò, Busetti & Granozzi, 1982); *N*-acetyl-Δ^Z-Phe-Gly-OH (Pieroni, Montagnoli, Fissi, Merlino & Ciardelli, 1975)], mostly *N*-acetylated, have

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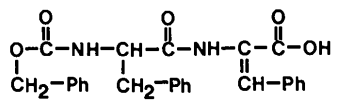
been studied by the X-ray method. In all but one structure (Nitz, Holt, Rubin & Stammer, 1981) the orientation around the Cα–Cβ double bond is *Z*, *i.e.* the substituent at Cβ is *trans* to the carbonyl group. The present paper reports the crystal structure of racemic *N*-[(phenylmethoxy)carbonyl]-phenylalanyl-Δ^2-phenylalanine (1).



(1)

Experimental. White needles from methanol, 0·16 × 0·19 × 0·55 mm; CAD-4, Mo $K\alpha$, graphite monochromator, lattice parameters from 25 reflections ($9 \le \theta \le 13°$). Data collection: 4986 reflections measured ($h_{max} = 8$, $k_{max} = \pm 16$, $l_{max} = \pm 15$, $\theta \le 27°$) in an $\omega/2\theta$ scan mode; three standard reflections monitored every 2 h, no significant change of intensity, 2806 observed [$I \ge 3\sigma(I_o)$], Lp correction, absorption neglected, space group $P\bar{1}$ (No. 2); direct methods (*MULTAN*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1981), anisotropic full-matrix least squares on *F*, H atoms (from ΔF synthesis) isotropic; final cycle 394 parameters $(\Delta/\sigma)_{max} = 0{\cdot}07$, $R = 0{\cdot}039$, $wR = 0{\cdot}047$, $S = 1{\cdot}50$, $w^{-1} = \sigma^2(I_o) + 0{\cdot}04|F|^2$; final ΔF shows $\delta_{max} = 0{\cdot}135$ e Å^{-3}.

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Programs used: *PARST* (Nardelli, 1983), CAD-4 *SDP* (Frenz, 1978) and *MULTAN*81 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1981). The final atomic parameters are given in Table 1.*

Discussion. The numbering scheme of atoms and a view of the molecule are shown in Fig. 1, while the structural parameters are given in Tables 2 and 3.

According to the torsion angles of the main chain and the mode of hydrogen bonding the structure of (1) can be classified as distorted or parallel β-sheet [$\varphi_1 = -89{\cdot}3$ (2), $\psi_1 = 114{\cdot}4$ (2)°] mixed with a left-handed α helix [$\varphi_2 = 50{\cdot}4$ (3), $\psi_2 = 21{\cdot}2$ (3)°], due to an unsaturated function in the second residue. Three other known uncharged oligopeptides containing diphenylalanyl fragments [Boc-Phe-Phe-OBz (Yamashita, Kato, Yamane & Ashida, 1982); Br-Ac-Phe-Phe-OEt and Cl-Ac-Phe-Phe-OEt (Wei, Doherty & Einstein, 1972)] form parallel β-sheet structures with φ and ψ angles closer to the predicted values of −119 and 113° (Schellman & Schellman, 1964) than those found in this study for the phenylalanyl residue. The side-chain conformation in Phe [$\chi_{1^1} = -177{\cdot}6$ (2), $\chi_{1^2} = 79{\cdot}9$ (2)°] is close to classical [180 and 90 ±30° (Benedetti, Morelli, Nemethy & Scheraga, 1983)] while $\chi_{2^1} = 2{\cdot}7$ (4) and $\chi_{2^2} = 16{\cdot}0$ (4)° for ΔPhe are due to the double C(18)–C(20) bond. The other known *X*-Phe-Phe-*Y* peptides [Gly-Phe-Phe (Précigoux, Cotrait & Geoffre, 1986); Tyr-Tyr (Cotrait, Bideau, Beurskens, Bosman & Beurskens, 1984); Phe-Phe-OEt (Yamashita, Kato, Yamane & Ashida, 1982) and D-Phe-Phe-OMe, (Pattabhi & Venkatesan, 1970)] exist in the crystals as zwitterions or are charged at the terminal N atom and they should not be compared with (1).

Table 1. *Fractional atomic coordinates* (×10⁴) *and equivalent isotropic temperature factors*

$B_{eq} = \frac{4}{3}(a^{*2}B_{11} + \ldots + b^*c^*B_{23}\cos\alpha^*)$.

	x	*y*	*z*	B_{eq}(Å²)
O(1)	−1528 (2)	7406 (1)	890 (1)	3·73 (3)
O(2)	−2816 (2)	6921 (1)	2170 (1)	3·92 (3)
O(3)	1253 (2)	4439 (1)	1200 (1)	3·35 (3)
O(4)	5530 (2)	5057 (1)	3304 (1)	4·42 (3)
O(5)	3580 (2)	6103 (1)	2722 (1)	3·90 (3)
N(1)	−2006 (2)	5722 (1)	829 (1)	2·89 (3)
N(2)	−55 (2)	4788 (1)	2666 (1)	2·77 (3)
C(1)	110 (4)	8969 (2)	2183 (2)	4·12 (5)
C(2)	1956 (4)	8532 (2)	2250 (2)	4·90 (6)
C(3)	3558 (4)	8993 (2)	2990 (2)	6·04 (7)
C(4)	3344 (5)	9894 (2)	3656 (2)	7·95 (8)
C(5)	1562 (6)	10346 (2)	3607 (3)	9·2 (1)
C(6)	−82 (5)	9875 (2)	2871 (2)	6·82 (8)
C(7)	−1636 (4)	8494 (2)	1372 (2)	4·44 (5)
C(8)	−2167 (3)	6690 (1)	1366 (1)	2·85 (4)
C(9)	−2319 (3)	4800 (1)	1214 (1)	2·72 (4)
C(10)	−3191 (3)	3814 (1)	385 (1)	3·43 (4)
C(11)	−3639 (3)	2837 (1)	742 (1)	3·35 (4)
C(12)	−2239 (4)	2109 (2)	654 (2)	5·09 (6)
C(13)	−2675 (4)	1212 (2)	961 (2)	6·93 (7)
C(14)	−4500 (5)	1013 (2)	1353 (2)	6·53 (7)
C(15)	−5894 (4)	1742 (2)	1473 (2)	5·55 (6)
C(16)	−5464 (3)	2646 (2)	1167 (2)	4·37 (5)
C(17)	−205 (3)	4658 (1)	1694 (1)	2·52 (4)
C(18)	1813 (3)	4690 (1)	3222 (1)	2·85 (4)
C(19)	3823 (3)	5290 (2)	3086 (1)	3·13 (4)
C(20)	1876 (3)	4151 (2)	3894 (1)	3·27 (4)
C(21)	223 (3)	3505 (1)	4225 (1)	3·25 (4)
C(22)	685 (3)	3257 (2)	5109 (1)	4·03 (5)
C(23)	−794 (4)	2684 (2)	5493 (2)	5·04 (6)
C(24)	−2766 (4)	2341 (2)	4994 (2)	5·54 (6)
C(25)	−3254 (4)	2544 (2)	4108 (2)	5·57 (6)
C(26)	−1771 (3)	3120 (2)	3718 (2)	4·57 (5)

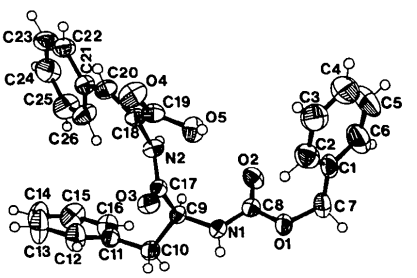


Fig. 1. A view of (1) (*ORTEP*, Johnson, 1976) and numbering of atoms.

* Lists of structure factors, H-atom parameters and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43850 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The hydrogen-bonding system consists of three hydrogen bonds. Two intermolecular hydrogen bonds, O(5)–H···O(2) ($x+1, y, z$) and N(2)–H···O(4) ($x-1, y, z$) join molecules of the same configuration into chains running along the x axis. The enantiomeric chains are paired into *RS* double chains by N(1)–H···O(3) hydrogen bonds. As a result the hydrophilic frames of (1), linked by six hydrogen bonds with three neighbouring molecules, form the backbone of the racemic double chains with phenyl substituents filling the space (Fig. 2).

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

O(1)–C(7)	1·439 (3)	C(9)–C(17)	1·524 (3)
O(1)–C(8)	1·340 (2)	C(10)–C(11)	1·509 (2)
O(2)–C(8)	1·219 (2)	C(11)–C(12)	1·379 (3)
O(3)–C(17)	1·227 (2)	C(11)–C(16)	1·383 (3)
O(4)–C(19)	1·210 (3)	C(12)–C(13)	1·371 (4)
O(5)–C(19)	1·324 (3)	C(13)–C(14)	1·361 (4)
N(1)–C(8)	1·332 (2)	C(14)–C(15)	1·377 (4)
N(1)–C(9)	1·457 (2)	C(15)–C(16)	1·378 (4)
N(2)–C(17)	1·337 (2)	C(18)–C(19)	1·488 (3)
N(2)–C(18)	1·421 (2)	C(18)–C(20)	1·327 (3)
C(1)–C(2)	1·383 (4)	C(20)–C(21)	1·465 (3)
C(1)–C(6)	1·363 (4)	C(21)–C(22)	1·391 (2)
C(1)–C(7)	1·499 (3)	C(21)–C(26)	1·393 (3)
C(2)–C(3)	1·377 (3)	C(22)–C(23)	1·376 (4)
C(3)–C(4)	1·347 (4)	C(23)–C(24)	1·370 (3)
C(4)–C(5)	1·355 (5)	C(24)–C(25)	1·369 (4)
C(5)–C(6)	1·393 (5)	C(25)–C(26)	1·385 (4)
C(9)–C(10)	1·532 (2)		

C(7)–O(1)–C(8)	116·9 (2)	C(12)–C(13)–C(14)	121·0 (3)
C(8)–N(1)–C(9)	122·3 (1)	C(13)–C(14)–C(15)	119·4 (3)
C(17)–N(2)–C(18)	123·0 (2)	C(14)–C(15)–C(16)	119·9 (3)
C(6)–C(1)–C(7)	119·7 (3)	C(11)–C(16)–C(15)	120·9 (2)
C(2)–C(1)–C(7)	122·2 (3)	N(2)–C(17)–C(9)	116·2 (2)
C(2)–C(1)–C(6)	118·1 (3)	O(3)–C(17)–C(9)	120·7 (1)
C(1)–C(2)–C(3)	121·3 (3)	O(3)–C(17)–N(2)	123·1 (2)
C(2)–C(3)–C(4)	119·6 (3)	N(2)–C(18)–C(20)	124·7 (2)
C(3)–C(4)–C(5)	120·6 (3)	N(2)–C(18)–C(19)	117·6 (1)
C(4)–C(5)–C(6)	120·2 (3)	C(19)–C(18)–C(20)	117·6 (2)
C(1)–C(6)–C(5)	120·2 (3)	O(5)–C(19)–C(18)	114·3 (2)
O(1)–C(7)–C(1)	113·2 (2)	O(4)–C(19)–C(18)	122·4 (2)
O(2)–C(8)–N(1)	126·5 (2)	O(4)–C(19)–O(5)	123·3 (2)
O(1)–C(8)–N(1)	110·2 (1)	C(18)–C(20)–C(21)	131·8 (2)
O(1)–C(8)–O(2)	123·3 (2)	C(20)–C(21)–C(26)	124·8 (2)
N(1)–C(9)–C(17)	108·4 (1)	C(20)–C(21)–C(22)	117·5 (2)
N(1)–C(9)–C(10)	110·4 (1)	C(22)–C(21)–C(26)	117·7 (2)
C(10)–C(9)–C(17)	110·2 (1)	C(21)–C(22)–C(23)	121·5 (2)
C(9)–C(10)–C(11)	113·1 (1)	C(22)–C(23)–C(24)	119·6 (2)
C(10)–C(11)–C(16)	121·0 (2)	C(23)–C(24)–C(25)	120·4 (3)
C(10)–C(11)–C(12)	120·9 (2)	C(24)–C(25)–C(26)	120·2 (3)
C(12)–C(11)–C(16)	118·1 (2)	C(21)–C(26)–C(25)	120·5 (2)
C(11)–C(12)–C(13)	120·6 (3)		

Table 3. *Selected torsion angles* (°) *and hydrogen-bond parameters*

O(1)–C(8)–N(1)–C(9)	(ω_1)	171·6 (2)	N(2)–C(18)–C(19)–O(5)	(ψ_2)	21·2 (3)
C(8)–N(1)–C(9)–C(17)	φ_1	−89·3 (2)	O(4)=C(19)–C(18)=C(20)		24·1 (3)
N(1)–C(9)–C(17)–N(2)	ψ_1	114·4 (2)	N(2)–C(18)=C(20)–C(21)		2·7 (4)
C(9)–C(17)–N(2)–C(18)	ω_2	−179·5 (2)	O(2)=C(8)–N(1)–C(9)		−9·2 (3)
C(17)–N(2)–C(18)–C(19)	φ_2	50·4 (3)	O(3)=C(17)–N(2)–C(18)		0·3 (3)

X–H···Y	X–H(Å)	X···Y(Å)	H···Y(Å)	X–H···Y(°)	Symmetry code for Y
O(5)–H···O(2)	0·93 (3)	2·695 (2)	1·76 (3)	174 (3)	$x+1, y, z$
N(1)–H···O(3)	0·87 (2)	2·907 (2)	2·05 (2)	170 (2)	$1-x, 1-y, -z$
N(2)–H···O(4)	0·86 (2)	3·070 (2)	2·24 (2)	163 (1)	$x-1, y, z$

The results support the observation of Aubry, Boussard & Marraud (1984) that incorporation of a dehydrophenylalanine into a peptide chain can disturb its structure and stabilize the β-turn. This is shown by the torsional angles $\varphi_1 = -89{\cdot}3$, $\psi_1 = 114{\cdot}4$, $\varphi_2 = 50{\cdot}4$ and $\psi_2 = 21{\cdot}2°$. The values are close to those observed in the type II β-turn of saturated peptides [−63, 128, 85 and 2° (Rose, Gierasch & Smith, 1985)]. Also the *inter*molecular hydrogen bond O(5)···O(2) is somewhat similar to the *intra*molecular hydrogen bond N($i+3$)···O(i) often found in type II β-bending (Venkatachalam, 1968). The differences result from the presence of a flat C(sp^2)α atom in the peptide backbone and two bulky C(α) substituents.

The C(18)–C(20) double bond in the dehydrophenylalanine residue has *syn* orientation towards the neighbouring C(19)–O(4) and *anti* orientation to the C(17)–O(3) carbonyl groups. The molecule maintains planarity of the whole dehydrophenylalanine residue through conjugation of benzene, C(18)=C(20) and C(19)=O(4) unsaturated systems. The torsion angles in the fragment are 16·0, 178·7 and 24·0° [C(22)–C(21)–C(20)=C(18), C(21)–C(20)=C(18)–C(19) and C(20)=C(18)–C(19)=O(4)]. The shortening of the C(20)–C(21), N(2)–C(18) and C(18)–C(19) bonds to 1·465 (3), 1·421 (2) and 1·488 (3) Å, respectively, is due to conjugation, the phenomenon commonly observed in α,β-unsaturated amino acids (Ajò, Bussetti & Granozzi, 1982). The abnormal values of the C(18)=C(20)–C(21) and C(20)–C(21)–C(26) angles [131·8 (2) and 124·8 (2)° respectively] result from steric repulsion between phenyl rings of Phe and ΔPhe (Fig. 1).

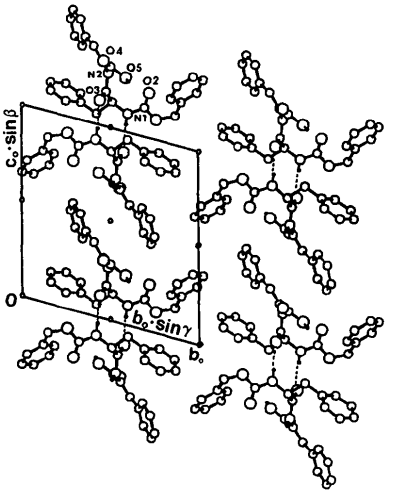


Fig. 2. Packing of molecules viewed along the x axis. Hydrogen atoms attached to C atoms have been omitted for clarity.

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2-Chlorobiphenyl-4′-carbonitrile

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Abstract. $C_{13}H_8ClN$, $M_r = 213{\cdot}7$, monoclinic, $P2_1/c$, $a = 6{\cdot}78$ (4), $b = 23{\cdot}51$ (5), $c = 7{\cdot}25$ (2) Å, $\beta = 115{\cdot}3$ (2)°, $V = 1045$ Å^3, $Z = 4$, $D_m = 1{\cdot}32$, $D_x = 1{\cdot}36$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 2{\cdot}8$ mm^{-1}, $F(000) = 440$, $T = 293$ K, $R = 0{\cdot}046$ for 843 observed densitometer-measured equi-inclination Weissenberg data. The average C–C bond in the phenyl rings is 1·374 Å. The molecule is non-planar; the angle between the phenyl rings is 52·1 (1)°; the C–Cl bond is 1·731 (5) Å; the C–C ≡ N bonds are 1·421 (8) and 1·133 (7) Å, the C–C bond making an angle of 1·94 (6)° with the phenyl plane.

Introduction. The structure determination of the title compound forms part of an investigation into liquid-crystal compounds and their chemical precursors.

Experimental. D_m measured by flotation in aqueous cadmium *n*-dodecatungstoborate. Pale-yellow opaque crystals used in data collection about **c** and **a** had dimensions of 0·09 × 0·07 × 0·27 and 0·16 × 0·08 × 0·14 mm, respectively. 2574 reflections measured by the SERC Microdensitometer Service, Daresbury Laboratory, from multiple-film photographs using Cu $K\alpha$ radiation, 1989 unique, $-8 \le h \le 7$; $0 \le k \le 28$; $0 \le l \le 7$; 843 unique observed reflections; $R_{int} = 0{\cdot}05$. Structure solved by Patterson synthesis and refined (on F) by full-matrix least squares with anisotropic thermal parameters for the non-H atoms, H-atom positions, initially obtained from a difference synthesis and placed at geometrically reasonable positions, refined with constrained C–H bond distances and isotropic thermal parameters, final $R = 0{\cdot}046$, $w = 1/[\sigma^2(F) + 0{\cdot}005\,F^2]$,

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