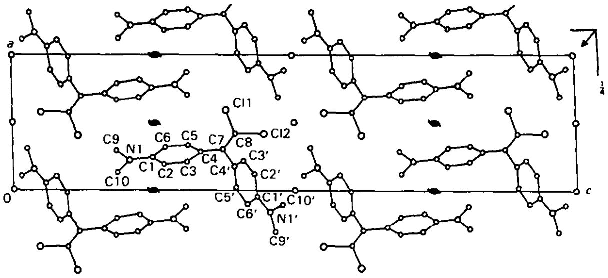

Fig. 2. The packing of the molecules as seen in projection down the *b* axis.

benzene derivatives. A similar effect has also been observed in *syn-p*-dimethylaminobenzaldehyde oxime (Bachechi & Zambonelli, 1972) and 2-(*p*-dimethylanilino)-4-phenyl-6a-thia-1,6-thiophthene (Hordvik & Saethre, 1972) which exemplified the effect of both π-donor and π-acceptor functional groups on the ring geometry (Domenicano, Vaciago & Coulson, 1975).

The molecular packing of the crystal viewed down the *b* axis is shown in Fig. 2. The structure is stabilized by van der Waals forces with no significant short intermolecular contacts.

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Structure of Benzyloxycarbonyl-L-alanyl-α-aminoisobutyric Acid, Z-L-Ala-Aib-OH

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Abstract. $C_{15}H_{20}N_2O_5$, $M_r = 308{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}545\,(1)$, $b = 10{\cdot}014\,(1)$, $c = 16{\cdot}692\,(1)$ Å, $V = 1595{\cdot}5\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}283$, $D_m = 1{\cdot}278$ Mg m^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}77$ mm^{-1}, $F(000) = 656$, $T = 295$ K, $R = 0{\cdot}047$ for 1508 reflections. The molecule has an extended form and no intramolecular hydrogen bonds. The α-aminoisobutyrate residue adopts a conformation close to that of an ideal right-handed 3_{10}-helix. The crystal structure is stabilized by three kinds of intermolecular hydrogen bonds.

Introduction. A number of short Aib-containing peptides studied so far adopt a β-turn conformation which characterizes a 3_{10}-helix (Prasad & Balaram, 1984). The conformation of the Aib residue is remarkably restricted owing to the bulky geminate methyl groups at the C$^\alpha$ atom. The torsion angles of the backbone at the Aib residue are close to the $\varphi = \pm 60$ and $\psi = \pm 30°$ angles of an ideal 3_{10}-helix. On the other hand, Ala residues are generally more flexible than the Aib residues, and the homo-oligomers have an extended conformation (Fawcett, Camerman & Camerman, 1975). However, the Ala residues in Aib-containing peptides tend to take torsion angles close to those of the 3_{10}-helix. As such, in an extreme example, Boc-L-Ala-Aib-OH (Bosh, Voges, Jung & Winter, 1983), the Ala residue has a conformation close to the 3_{10}-helix, although the Aib conformation is different. Thus, the structure of the

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Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2)

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
C(1)	−0·3263 (4)	0·1739 (4)	0·2545 (3)	6·5 (2)
C(2)	−0·3700 (6)	0·1588 (6)	0·3329 (3)	8·8 (3)
C(3)	−0·4243 (5)	0·0406 (8)	0·3555 (3)	9·6 (3)
C(4)	−0·4360 (4)	−0·0635 (5)	0·3030 (3)	9·9 (3)
C(5)	−0·3917 (4)	−0·0483 (4)	0·2267 (3)	6·1 (2)
C(6)	−0·3365 (3)	0·0700 (3)	0·2009 (2)	4·3 (1)
C(7)	−0·2854 (4)	0·0872 (6)	0·1170 (2)	7·6 (2)
C(8)	−0·0464 (3)	0·0917 (3)	0·0720 (2)	3·6 (1)
C(9)	0·2018 (3)	0·0704 (3)	0·0395 (2)	3·5 (1)
C(10)	0·3089 (4)	0·0961 (4)	0·1046 (2)	5·8 (2)
C(11)	0·2605 (2)	−0·0278 (2)	−0·0222 (1)	3·0 (1)
C(12)	0·4329 (3)	−0·0459 (3)	−0·1325 (1)	3·6 (1)
C(13)	0·5440 (4)	0·0498 (3)	−0·1657 (2)	5·6 (1)
C(14)	0·3341 (4)	−0·0873 (4)	−0·1989 (2)	5·7 (2)
C(15)	0·5109 (3)	−0·1674 (3)	−0·1003 (2)	3·5 (1)
N(1)	0·0715 (2)	0·0209 (2)	0·0726 (1)	3·7 (1)
N(2)	0·3570 (2)	0·0253 (2)	−0·0696 (1)	3·3 (1)
O(1)	−0·1457 (2)	0·0277 (3)	0·1128 (2)	6·1 (1)
O(2)	−0·0630 (3)	0·1986 (2)	0·0396 (1)	5·4 (1)
O(3)	0·2252 (2)	−0·1460 (2)	−0·0254 (1)	4·1 (1)
O(4)	0·5371 (3)	−0·2623 (2)	−0·1424 (1)	4·8 (1)
O(5)	0·5559 (2)	−0·1558 (2)	−0·0260 (1)	4·6 (1)

Table 2. *Bond distances* (Å) *and bond angles* (°)

C(1)—C(2)	1·382 (7)	C(1)—C(6)	1·376 (5)
C(2)—C(3)	1·346 (9)	C(3)—C(4)	1·366 (9)
C(4)—C(5)	1·351 (7)	C(5)—C(6)	1·366 (5)
C(6)—C(7)	1·493 (5)	C(7)—O(1)	1·462 (5)
C(8)—N(1)	1·330 (3)	C(8)—O(1)	1·332 (4)
C(8)—O(2)	1·210 (4)	C(9)—C(10)	1·514 (5)
C(9)—C(11)	1·530 (4)	C(9)—N(1)	1·448 (4)
C(11)—N(2)	1·326 (3)	C(11)—O(3)	1·232 (3)
C(12)—C(13)	1·533 (4)	C(12)—C(14)	1·513 (4)
C(12)—C(15)	1·524 (4)	C(12)—N(2)	1·461 (3)
C(15)—O(4)	1·208 (4)	C(15)—O(5)	1·318 (4)
C(2)—C(1)—C(6)	120·8 (4)	C(1)—C(2)—C(3)	118·5 (5)
C(2)—C(3)—C(4)	121·5 (5)	C(3)—C(4)—C(5)	119·6 (5)
C(4)—C(5)—C(6)	121·0 (4)	C(1)—C(6)—C(5)	118·6 (4)
C(1)—C(6)—C(7)	120·0 (4)	C(5)—C(6)—C(7)	121·4 (4)
C(6)—C(7)—O(1)	107·2 (3)	N(1)—C(8)—O(1)	110·0 (3)
N(1)—C(8)—O(2)	125·9 (3)	O(1)—C(8)—O(2)	124·1 (3)
C(10)—C(9)—C(11)	110·2 (2)	C(10)—C(9)—N(1)	111·4 (3)
C(11)—C(9)—N(1)	110·6 (2)	C(9)—C(11)—N(2)	113·5 (2)
C(9)—C(11)—O(3)	123·1 (2)	N(2)—C(11)—O(3)	123·3 (2)
C(13)—C(12)—C(14)	109·7 (2)	C(13)—C(12)—C(15)	106·8 (2)
C(13)—C(12)—N(2)	107·3 (2)	C(14)—C(12)—C(15)	110·1 (3)
C(14)—C(12)—N(2)	110·5 (2)	C(15)—C(12)—N(2)	112·2 (2)
C(12)—C(15)—O(4)	121·6 (3)	C(12)—C(15)—O(5)	114·9 (2)
O(4)—C(15)—O(5)	123·3 (3)	C(8)—N(1)—C(9)	122·8 (2)
C(11)—N(2)—C(12)	125·3 (2)	C(7)—O(1)—C(8)	118·5 (3)

title dipeptide, which has the same L-Ala-Aib-OH sequence, was determined in order to obtain further information about the Ala and Aib conformations.

Experimental. Z-L-Ala-Aib-OH crystals from an ethyl acetate and diethyl ether solution as colorless prisms; density by flotation; crystal dimensions 0·2 × 0·3 × 0·3 mm; Rigaku AFC5-RU diffractometer with graphite monochromator; unit-cell parameters from 20 reflections ($30 < 2\theta < 54°$); 1540 reflections measured with $2\theta < 120°$; $0 \le h \le 11$, $0 \le k \le 11$, $0 \le l \le 19$; 2θ–ω scan; no significant intensity fluctuation for three standard reflections monitored every 56 reflections; intensities corrected for Lorentz and polarization effects, but not for absorption and extinction; 1508 reflections with $F_o > 3\sigma(F_o)$ used for structure determination. Structure solved with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); refined by block-diagonal least-squares, $\sum w(|F_o| - |F_c|)^2$ minimized; $w = [\sigma^2(F_o) + (0{\cdot}023F_o)^2]^{-1}$; non-H atoms anisotropic; H atoms from difference map, isotropic; $R = 0{\cdot}047$, $wR = 0{\cdot}067$, $S = 1{\cdot}70$, $(\Delta/\sigma)_{\max} = 0{\cdot}29$, $\Delta\rho_{\max} = 0{\cdot}16$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); computations performed on a FACOM M382 using *KPPXRAY* (Taga, Higashi & Iizuka, 1985).

Discussion. The final atomic coordinates and thermal parameters for non-H atoms of Z-L-Ala-Aib-OH are listed in Table 1.* Bond distances and angles are listed in Table 2. A perspective view of the molecule is shown in Fig. 1.

The bond distances and angles show no significant deviations from the usual values. However, the

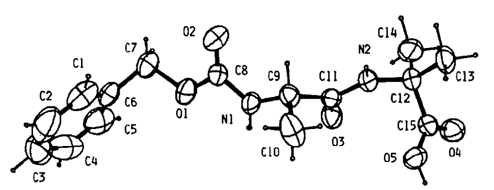

Fig. 1. A perspective view of the molecule with atomic numbering.

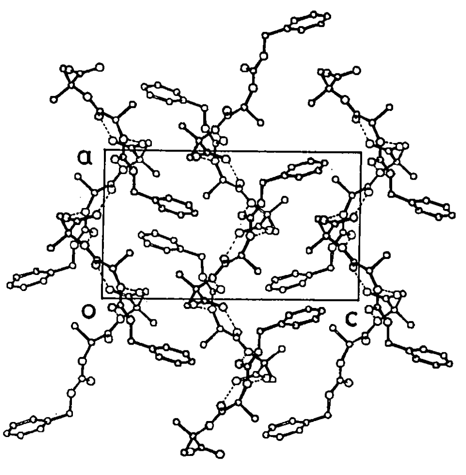

Fig. 2. Packing of the molecules viewed down **b**. The intermolecular hydrogen bonds are indicated as dashed lines.

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* Lists of anisotropic thermal parameters for non-H atoms, coordinates and isotropic thermal parameters for H atoms, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52151 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

C(12)—C(13) bond distance of 1·533 (4) Å is significantly longer than the C(12)—C(14) bond distance of 1·513 (4) Å, and the bond angles 110·5 (2)° for N(2)—C(12)—C(14) and 110·1 (3)° for C(15)—C(12)—C(14) are larger than 107·3 (2)° for N(2)—C(12)—C(13) and 106·8 (2)° for C(15)—C(12)—C(13). Such asymmetry around the C^{α} atom of the Aib residue has been observed in other Aib-containing peptides. According to Paterson, Rumsey, Benedetti, Némethy & Scheraga (1981), the asymmetric geometries are sensitive to the backbone torsion angles of the Aib residue, and have a correlation with the helical conformation type. The present Aib residue has the torsion angles $\varphi_2 = -57{\cdot}2$ (2), $\psi_2 = -31{\cdot}4$ (3) and $\omega_2 = 178{\cdot}5$ (2)°, which are close to $\varphi = -60$, $\psi = -30$ and $\omega = 180°$ for an ideal right-handed 3_{10}-helix. The asymmetric covalent-bond geometry for the torsion angles corresponding to the right-handed helix is consistent with other data (Benedetti, Bavoso, Blasio, Pavone, Pedone, Crisma, Bonora & Toniolo, 1982). The Ala residue has the torsion angles $\varphi_1 = -125{\cdot}5$ (2), $\psi_1 = 163{\cdot}1$ (2) and $\omega_1 = 185{\cdot}8$ (2)°. The values are close to $\varphi = -113$ and $\psi = 165°$ in L-alanyl-L-alanine (Fletterick, Tsai & Hughes, 1971). The torsion angles for the N-terminal protected group are −178·0 (3)° for N(1)—C(8)—O(1)—C(7), −138·3 (3)° for C(8)—O(1)—C(7)—C(6) and −80·0 (3)° for O(1)—C(7)—C(6)—C(5). The molecular conformation differs from that of Boc-L-Ala-Aib-OH ($\varphi_1 = -66$, $\psi_1 = -24$, $\varphi_2 = -175$ and $\psi_2 = -170°$), and also differs from that of a similar C-terminal unprotected dipeptide Z-Aib-Aib-OH ($\varphi_1 = -65$, $\psi_1 = -26$, $\varphi_2 = 50$ and $\psi_2 = 45°$) (Benedetti, Pedone & Toniolo, 1980). Since the molecule has no intramolecular hydrogen bonds, the molecular conformation largely depends on intermolecular packing forces.

The crystal structure is shown in Fig. 2. The molecules are packed by three kinds of intermolecular hydrogen bonds, N(1)—H···O(4)($-\frac{1}{2}+x$, $-\frac{1}{2}-y$, $-z$) 2·859 (3), N(2)—H···O(2)($\frac{1}{2}+x$, $\frac{1}{2}-y$, $-z$) 2·912 (3), and O(5)—H···O(3)($-\frac{1}{2}+x$, $-\frac{1}{2}-y$, $-z$) 2·699 (3) Å. The hydrogen-bonded molecules form a layer perpendicular to the *c* axis. The molecules of the adjacent layers are close to each other with the usual van der Waals distances, the short intermolecular distances being 3·554 (5) Å between C(10) and C(14)($\frac{1}{2}-x$, $-y$, $\frac{1}{2}+z$) and 3·368 (6) Å between C(2) and O(4)($\frac{1}{2}-x$, $-y$, $\frac{1}{2}+z$).

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Structure of 8-Ethyl-9-methyladenine Dihydrate

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Abstract. $C_8H_{11}N_5.2H_2O$, $M_r = 213{\cdot}2$, triclinic, $P\bar{1}$, $a = 7{\cdot}460$ (3), $b = 7{\cdot}824$ (2), $c = 9{\cdot}929$ (3) Å, $\alpha = 112{\cdot}28$ (2), $\beta = 102{\cdot}48$ (3), $\gamma = 89{\cdot}06$ (3)°, $V = 522{\cdot}3$ (3) Å^3, $Z = 2$, $D_x = 1{\cdot}36$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·12 mm^{-1}, $F(000) = 228$, $T = 105$ K, final $R = 0{\cdot}083$, $wR = 0{\cdot}067$ for 1337

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