The central C(1)—C(1′) bond length, 1.591 Å, is slightly longer in this structure than in compound (2). This is not unexpected as the ring size is now larger. The greater steric congestion is also clearly reflected in the values of the bond angles around the two central C atoms. The six C—C—C bond angles within the molecule are smaller than the tetrahedral value while the C—C—C bond angles involving the central C atoms are greater. These deviations are consistent with the existence of steric congestion in the molecule.

Comparison with the dinitro analogues of compounds (1)–(3). All the compounds (1)–(3) are in the *trans* conformation with the N(1)—C(1N)—C(1) bond angle lying in the range 176.3–178.5°. The cyclopentyl ring adopts the 'envelope' structure while the cyclohexyl and cycloheptyl rings are in the chair conformation. The structures of (1) and (3) belong to the C_{2h} point group while that of (2) is of approximate C_{2h} symmetry. The central C(1)—C(1′) bond length increases with ring size as a result of increasing steric congestion.

On the other hand, the shape of the polar substituent in the dinitro analogues is angular rather than cylindrical. As expected, this causes greater steric congestion. These dinitro bicycloalkanes adopt *gauche* conformations with the nitro groups in close proximity. As the ring size increases, the strain arising from contacts between the nitro groups increases and is reflected in increasing inter-ring and C—N bond lengths. The cyclopentyl ring adopts a skewed structure rather than the 'envelope' form. The cyclohexyl and cycloheptyl rings adopt chair conformations, as in compounds (2) and (3), but are of lower symmetry.

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Structure of *N-tert*-Butyloxycarbonyl-α-aminoisobutyryl-DL-pipecolyl-α-aminoisobutyric Acid Methyl Ester*

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Abstract. $C_{20}H_{35}N_3O_6$ (Boc-Aib-DL-Pip-Aib-OMe, Boc = *tert*-butyloxycarbonyl, Aib = α-aminoisobutyric acid, Pip = pipecolic acid, OMe = methoxy), M_r = 413.5, monoclinic, $P2_1/c$, a = 18.055 (3), b = 15.048 (3), c = 17.173 (3) Å, β = 91.7 (1)°, V = 4663.8 (9) Å^3, Z = 8, D_m = 1.16, D_x = 1.178 Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, μ = 0.081 mm^{-1}, $F(000)$ = 1792, T = 297 K. The final R value for 4925 [$I \geq 3\sigma(I)$] reflections is 0.065 (wR = 0.067). The peptide backbone of the two independent molecules in the asymmetric unit is folded at the -Aib-Pip- sequence to form a type-I (I′) β-bend stabilized by a 1←4 intramolecular N—H···O=C hydrogen bond between the Aib(3) peptide N—H and Boc urethane C=O groups.

Introduction. Pipecolic acid, also referred to as homoproline or piperidine-2-carboxylic acid, is one

* Linear Oligopeptides. 262. Part 261: Bardi, Piazzesi, Crisma, Toniolo, Sukumar & Balaram (1992).

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for the non-*H *atoms of Boc-Aib-*DL*-Pip-Aib-*OMe *with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	U_{eq}
Molecule *A*				
O(1)	6257 (1)	1372 (2)	8821 (2)	64 (1)
O(2)	5109 (1)	760 (2)	8813 (2)	60 (1)
O(3)	4425 (1)	−1096 (2)	7911 (2)	69 (1)
O(4)	2566 (1)	275 (2)	7550 (2)	64 (1)
O(5)	1771 (1)	1350 (2)	8856 (2)	75 (1)
O(6)	2527 (1)	2154 (2)	8120 (2)	70 (1)
N(1)	6019 (1)	241 (2)	8051 (2)	50 (1)
N(2)	4527 (1)	121 (2)	7176 (2)	48 (1)
N(3)	3537 (1)	792 (2)	8256 (2)	48 (1)
C(1)	5823 (3)	1793 (3)	10105 (3)	81 (2)
C(2)	6816 (3)	2580 (4)	9430 (3)	96 (2)
C(3)	5526 (2)	2719 (3)	8929 (3)	74 (2)
C(4)	6082 (2)	2124 (3)	9329 (2)	58 (2)
C(5)	5737 (2)	783 (3)	8586 (2)	48 (1)
C(6)	5660 (2)	−591 (3)	7818 (2)	51 (1)
C(7)	5799 (3)	−1271 (3)	8458 (3)	96 (2)
C(8)	6014 (2)	−909 (3)	7073 (3)	76 (2)
C(9)	4821 (2)	−519 (3)	7658 (2)	47 (1)
C(10)	3726 (2)	40 (3)	7010 (2)	60 (1)
C(11)	3504 (3)	351 (5)	6222 (3)	121 (3)
C(12)†	3792 (4)	1119 (5)	6019 (4)	84 (2)§
C(12′)‡	4078 (8)	735 (10)	5592 (9)	47 (4)§
C(13)	4701 (3)	1054 (4)	6031 (3)	117 (2)
C(14)	4901 (2)	894 (3)	6867 (3)	68 (2)
C(15)	3236 (2)	383 (3)	7634 (2)	46 (1)
C(16)	3087 (2)	1059 (3)	8917 (2)	50 (1)
C(17)	2901 (3)	260 (3)	9413 (3)	85 (2)
C(18)	3526 (2)	1756 (3)	9394 (3)	67 (2)
C(19)	2373 (2)	1506 (3)	8625 (2)	52 (1)
C(20)	1890 (3)	2657 (4)	7842 (3)	104 (2)
Molecule *B*				
O(1)′	11159 (1)	3796 (2)	1204 (2)	58 (1)
O(2)′	10042 (1)	4483 (2)	1295 (2)	57 (1)
O(3)′	9507 (1)	6241 (2)	2314 (2)	65 (1)
O(4)′	7559 (1)	4768 (2)	2626 (2)	67 (1)
O(5)′	6715 (1)	3751 (2)	1216 (2)	72 (1)
O(6)′	7587 (1)	2967 (2)	1848 (2)	73 (1)
N(1)′	11020 (1)	4809 (2)	2104 (2)	49 (1)
N(2)′	9550 (1)	4946 (2)	2959 (2)	46 (1)
N(3)′	8482 (1)	4444 (2)	1830 (2)	55 (1)
C(1)′	10585 (2)	3563 (3)	−98 (2)	73 (2)
C(2)′	11624 (3)	2648 (3)	478 (3)	89 (2)
C(3)′	10362 (2)	2517 (3)	1005 (3)	73 (2)
C(4)′	10906 (2)	3127 (3)	632 (2)	56 (2)
C(5)′	10689 (2)	4378 (3)	1505 (2)	46 (1)
C(6)′	10718 (2)	5627 (3)	2433 (2)	50 (1)
C(7)′	10899 (2)	6401 (3)	1898 (3)	74 (2)
C(8)′	11096 (2)	5779 (3)	3237 (3)	65 (2)
C(9)′	9876 (2)	5603 (3)	2543 (2)	46 (1)
C(10)′	8764 (2)	5073 (3)	3129 (2)	57 (1)
C(11)′	8584 (3)	4740 (5)	3926 (3)	96 (3)
C(12)′	8870 (3)	3835 (4)	4104 (3)	108 (3)
C(13)′	9699 (3)	3810 (4)	3984 (3)	91 (2)
C(14)′	9879 (2)	4090 (3)	3171 (3)	35 (1)
C(15)′	8223 (2)	4732 (3)	2501 (2)	49 (1)
C(16)′	7974 (2)	4242 (3)	1166 (2)	61 (1)
C(17)′	7663 (2)	5094 (4)	821 (3)	90 (2)
C(18)′	8412 (2)	3733 (4)	571 (3)	90 (2)
C(19)′	7345 (2)	3648 (3)	1429 (2)	57 (1)
C(20)′	7023 (3)	2355 (3)	2092 (3)	89 (2)

† Population parameter 0.75.
‡ Population parameter 0.25.
§ Refined isotropically.

of the few ring homologues of the conformationally restricted proline residue that have been incorporated into analogues of bioactive peptides (bradykinin, angiotensin II, TRH, oxytocin, MIF, ACE inhibitor, thrombin substrate and inhibitor, collagen model). Also, the presence of Pip residues characterizes the sequence of the highly hydrophobic Aib-rich peptide antibiotics efrapeptins and elvapeptins, potent inhibitors of ATPases activity, and the structure of the immunosuppressive agents FK 506 and rapamycin.

We describe here the results of the X-ray diffraction analysis of the fully protected tripeptide Boc-Aib-Pip-Aib-OMe, the synthesis and characterization of which have been reported by Sukumar (1987), representing the sequences 2–4 and 10–12 of efrapeptin D (Bullough, Jackson, Henderson, Cottee, Beechey & Linnett, 1982).

Experimental. Colourless crystals ($0.4 \times 0.3 \times 0.6$ mm) of Boc-Aib-DL-Pip-Aib-OMe were obtained from a chloroform solution by slow evaporation. D_m was measured by flotation. X-ray diffraction data were collected on a Philips PW 1100 four-circle diffractometer using Mo $K\alpha$ radiation (graphite monochromated). Accurate unit-cell parameters and crystal-orientation matrices (with e.s.d.'s) were determined from least-squares refinement of the 2θ, ω, χ and φ values of 25 carefully centred reflections with $8 < \theta < 15°$; θ–2θ scan, scan speed $0.02° s^{-1}$, scan width 1.2°. The h, k, l ranges measured were −21 to 21, 0 to 17, 0 to 20, respectively. Three standard reflections (424, 135, 225) were measured every 180 min and showed 5% variation in intensity. 8176 unique reflections were obtained to $\theta = 25°$, 4925 with $I \geq 3\sigma(I)$. Intensities were corrected for Lorentz and polarization effects, but not for absorption. The structure was solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and refined by conventional least-squares procedures. There are two molecules in the asymmetric unit (*A* and *B*). For molecule *A* disorder at the Pip residue was observed, and population parameters were applied to the C(12) and C(12′) atoms. Positional and isotropic thermal parameters refined smoothly. H atoms were placed in calculated positions [except for C(12) and C(12′)] and refined in the last cycle. 794 parameters were refined. Calculations were carried out using the *SHELX*76 program (Sheldrick, 1976). The quantity minimized was $\sum w(|F_o| - |F_c|)^2$, with $w = [\sigma^2|F| + 0.0006|F|^2]^{-1}$; final $R = 0.065$ ($wR = 0.067$); $(\Delta/\sigma)_{max} = 0.9$; $-0.4 < \Delta\rho < 0.4$ e $Å^{-3}$. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71–103). Table 1 gives the final atomic coordinates and equivalent isotropic thermal parameters for the non-H atoms.*

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55082 (34 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0294]

Discussion. A view of the molecule with the atom numbering is shown in Fig. 1. Bond lengths, bond angles and selected torsion angles are given in Table 2.

The values of bond lengths and bond angles are in agreement with literature data on the geometry of Aib (Prasad & Balaram, 1984; Valle, Crisma, Formaggio, Toniolo & Jung, 1987) and Pip (Rae, Raston & White, 1980) residues, *trans* peptide units (Benedetti, 1982), and Boc urethane derivatives (Benedetti, Pedone, Toniolo, Némethy, Pottle & Scheraga, 1980).

Molecules *A* and *B* have very similar conformations and are related by an approximate twofold axis parallel to *a* and passing through 0, $\frac{1}{4}$, 0. The C(4)—O(1) bond is in the usual *trans* arrangement relative to the C(5)—N(1) bond; this feature, accompanied by the *trans* conformation of the —CONH— group, allows one to classify the urethane moiety of these two molecules as type *b* (Benedetti *et al.*, 1980). The —OMe ester moiety adopts a conformation with respect to the C(16)—N(3) bond intermediate between the anticlinal and antiperiplanar arrangements (Dunitz & Strickler, 1968).

The tripeptide adopts a type-I (I′) β-bend conformation with Aib(1) and Pip(2) as the $i+1$ and $i+2$ corner residues, respectively. According to conformational energy calculations (Toniolo, Bardi, Piazzesi, Crisma, Balaram, Sukumar & Paul, 1989) the $i+2$ position of a type-I (I′) β-bend is available for a Pip residue, although with some distortion from its preferred set of backbone torsion angles. The 1←4 intramolecular hydrogen bond is seen between the Aib(3) peptide N—H and Boc urethane C=O groups, with an N···O distance of 2.97 (molecule *A*) and 2.99 Å (molecule *B*). Also the Aib(3) residue adopts a conformation in the helical region of the φ, ψ map, as Aib(1), but the helix handedness is opposite to that exhibited by the preceding residues. This feature has been consistently observed in the crystal structures of Aib-containing peptides

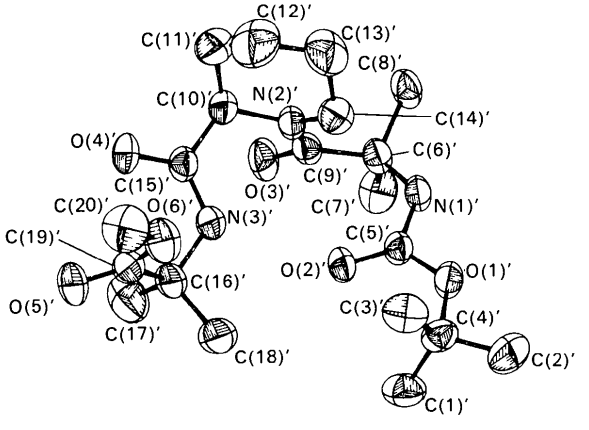

Fig. 1. Molecular structure of Boc-Aib-Pip-Aib-OMe (molecule *B*) with the numbering of atoms (ellipsoids at 50% probability).

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°) *for Boc-Aib-*DL*-Pip-Aib-OMe with e.s.d.'s in parentheses*

	Molecule *A*	Molecule *B*
C(1)—C(4)	1.510 (6)	1.515 (5)
C(2)—C(4)	1.498 (7)	1.513 (6)
C(3)—C(4)	1.497 (6)	1.501 (6)
C(4)—O(1)	1.469 (5)	1.470 (5)
O(1)—C(5)	1.344 (5)	1.334 (5)
C(5)—O(2)	1.210 (4)	1.223 (4)
C(5)—N(1)	1.340 (5)	1.342 (5)
N(1)—C(6)	1.460 (5)	1.466 (4)
C(6)—C(7)	1.517 (6)	1.525 (6)
C(6)—C(8)	1.524 (6)	1.539 (6)
C(6)—C(9)	1.535 (5)	1.538 (5)
C(9)—O(3)	1.213 (5)	1.227 (5)
C(9)—N(2)	1.367 (5)	1.363 (5)
N(2)—C(10)	1.471 (4)	1.470 (4)
C(10)—C(11)	1.476 (6)	1.502 (6)
C(11)—C(12)	1.318 (10)	1.485 (9)
C(11)—C(12′)	1.626 (16)	
C(12)—C(13)	1.644 (9)	1.517 (8)
C(12′)—C(13)	1.419 (15)	
C(13)—C(14)	1.490 (7)	1.503 (7)
C(14)—N(2)	1.453 (5)	1.460 (5)
C(10)—C(15)	1.501 (5)	1.523 (5)
C(15)—O(4)	1.225 (4)	1.225 (4)
C(15)—N(3)	1.335 (4)	1.329 (5)
N(3)—C(16)	1.471 (5)	1.474 (5)
C(1)—C(4)—C(2)	110.1 (5)	111.7 (4)
C(1)—C(4)—C(3)	112.4 (6)	112.2 (6)
C(2)—C(4)—C(3)	111.0 (4)	110.9 (4)
C(1)—C(4)—O(1)	110.3 (3)	111.1 (3)
C(2)—C(4)—O(1)	102.5 (5)	101.0 (5)
C(3)—C(4)—O(1)	109.9 (4)	109.1 (3)
C(4)—O(1)—C(5)	121.7 (5)	121.3 (6)
O(1)—C(5)—O(2)	125.1 (4)	125.7 (4)
O(1)—C(5)—N(1)	109.3 (6)	109.9 (6)
O(2)—C(5)—N(1)	125.6 (6)	124.3 (6)
C(5)—N(1)—C(6)	122.3 (5)	122.6 (5)
N(1)—C(6)—C(8)	107.8 (4)	108.1 (3)
N(1)—C(6)—C(7)	108.5 (4)	108.7 (3)
N(1)—C(6)—C(9)	114.6 (4)	114.0 (4)
C(8)—C(6)—C(9)	107.9 (4)	107.9 (4)
C(7)—C(6)—C(9)	108.6 (5)	108.8 (4)
C(7)—C(6)—C(8)	109.4 (4)	109.2 (3)
C(6)—C(9)—O(3)	118.3 (4)	118.1 (4)
C(6)—C(9)—N(2)	121.4 (4)	121.6 (4)
O(3)—C(9)—N(2)	119.9 (7)	119.8 (7)
C(9)—N(2)—C(10)	114.9 (4)	116.3 (4)
N(2)—C(10)—C(11)	113.0 (6)	111.9 (6)
C(10)—C(11)—C(12)	114.9 (6)	114.2 (5)
C(10)—C(11)—C(12′)	124.1 (8)	
C(11)—C(12)—C(13)	110.2 (7)	109.5 (6)
C(11)—C(12′)—C(13)	106.1 (9)	
C(12)—C(13)—C(14)	103.6 (7)	111.1 (7)
C(12′)—C(13)—C(14)	128.5 (9)	
C(13)—C(14)—N(2)	112.3 (4)	112.4 (4)
C(14)—N(2)—C(9)	127.5 (6)	126.3 (6)
C(14)—N(2)—C(10)	117.4 (4)	116.9 (5)
N(2)—C(10)—C(15)	115.4 (4)	114.7 (4)
C(11)—C(10)—C(15)	113.3 (4)	112.4 (5)
C(10)—C(15)—O(4)	118.1 (4)	118.1 (4)
C(10)—C(15)—N(3)	119.7 (6)	119.3 (6)
O(4)—C(15)—N(3)	122.2 (6)	122.5 (6)
C(15)—N(3)—C(16)	121.5 (6)	120.7 (6)
O(1)—C(5)—N(1)—C(6)	165.2 (6)*	164.9 (6)
C(5)—N(1)—C(6)—C(9)	45.8 (8)	43.6 (8)
N(1)—C(6)—C(9)—N(2)	50.4 (8)	54.6 (8)
C(9)—N(2)—C(10)—C(15)	79.8 (7)	87.5 (7)
C(14)—N(2)—C(10)—C(15)	−95.5 (7)	−85.5 (7)
N(2)—C(10)—C(15)—N(3)	5.1 (9)	−7.2 (9)
C(10)—C(15)—N(3)—C(16)	−173.8 (6)	−170.3 (6)
C(15)—N(3)—C(16)—C(19)	−45.4 (8)	−49.3 (8)
N(3)—C(16)—C(19)—O(5)	134.0 (7)	138.3 (7)
N(3)—C(16)—C(19)—O(6)	−51.3 (7)	−48.0 (7)

* Only values for the D enantiomer are given.

(Toniolo, Bonora, Bavoso, Benedetti, Di Blasio, Pavone & Pedone, 1983; Prasad & Balaram, 1984).

Both molecules *A* and *B* show the piperidine ring in a chair ($^4C_1$) conformation and the —CONH— substituent in the axial disposition, as expected from calculations (Toniolo *et al.*, 1989). The urethane and amide groups have the *trans* orientation, but the former is markedly out-of-plane. Interestingly, the tripeptide analogue *Z*-Aib-L-Pro-Aib-OMe is also folded in the crystal state, showing the same type of β-bend (distorted type-I) (Benedetti, Bavoso, Di Blasio, Pavone, Pedone, Toniolo, Bonora & Crisma, 1983).

In the crystal packing intermolecular N—H···O=C hydrogen bonds occur, involving the urethane Aib(1) N—H as donor and the peptide Pip(2) C=O as acceptor. Alternating *A* and *B* molecules form infinite chains along *a*. The N(1) atom of an *A* molecule is hydrogen bonded to the O(4′) atom of an enantiomeric *B* molecule (x, $\frac{1}{2}-y$, $\frac{1}{2}+z$), while the N(1′) atom of a *B* molecule is hydrogen bonded to the O(4) atom of an enantiomeric *A* molecule ($x+1$, $\frac{1}{2}-y$, $z-\frac{1}{2}$). The corresponding N···O distances are 2.89 and 2.88 Å.

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Structure of Tetrachlorophosphonium Hexafluoroantimonate

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Abstract. $[PCl_4][SbF_6]$, $M_r = 408.526$, tetragonal, $P4/n$, $a = 8.568$ (3), $c = 6.531$ (5) Å, $V = 479.4$ Å^3, $Z = 2$, $D_x = 2.830$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 4.22$ mm^{-1}, $F(000) = 376$, $T = 169$ K, final $R = 0.059$ for 425 unique observed [$F \geq 4.0\sigma(F)$] diffractometer data. The unit cell contains two $[PCl_4]^+$ cations [P—Cl 1.929 (2) Å (×4), Cl—P—Cl 109.15 (8) (×2), 109.63 (8)° (×4)] and two $[SbF_6]^-$ anions [Sb—F 1.864 (5) (×4), 1.924 (9) (×1), 1.912 (8) Å (×1), F—Sb—F 90.0 or 180.0°]. The anion resides on a fourfold axis and the cation on a fourfold inversion axis.

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