C2*c*	−0.0827 (3)	0.4824 (3)	0.1481 (3)	0.050 (1)
C3*c*	−0.1241 (3)	0.3758 (3)	0.0163 (3)	0.054 (1)
C4*c*	−0.0455 (3)	0.2702 (3)	−0.0119 (3)	0.052 (1)
C5*c*	0.0722 (3)	0.2694 (3)	0.0900 (3)	0.056 (1)
C6*c*	0.1107 (3)	0.3779 (3)	0.2215 (3)	0.054 (1)

Table 4. *Geometric parameters* (Å, °) *for* 24MOPD

O1—C5	1.425 (4)	N2—C4	1.138 (4)
O1—C2*c*	1.370 (4)	C1—C1*a*	1.488 (5)
O2—C6	1.437 (4)	C1—C1*b*	1.490 (3)
O2—C4*c*	1.374 (3)	C2—C3	1.357 (4)
N1—C1	1.287 (3)	C2—C4	1.450 (4)
N1—C2	1.399 (4)	C3—C1*c*	1.448 (3)
C5—O1—C2*c*	118.6 (2)	C3—C2—C4	116.2 (3)
C6—O2—C4*c*	117.7 (2)	C2—C3—C1*c*	128.3 (3)
C1—N1—C2	123.1 (2)	N2—C4—C2	177.1 (3)
N1—C1—C1*a*	116.5 (2)	O1—C2*c*—C1*c*	114.9 (2)
N1—C1—C1*b*	125.1 (3)	O1—C2*c*—C3*c*	122.8 (3)
C1*a*—C1—C1*b*	118.4 (2)	O2—C4*c*—C5*c*	124.1 (3)
N1—C2—C3	122.8 (2)	C3*c*—C4*c*—C5*c*	121.4 (2)
N1—C2—C4	120.4 (3)		
C2—N1—C1—C1*a*	173.5 (3)	N1—C1—C1*b*—C2*b*	118.2 (4)
C2—N1—C1—C1*b*	−7.9 (5)	N1—C2—C3—C1*c*	−7.4 (5)
C1—N1—C2—C3	151.6 (3)	C4—C2—C3—C1*c*	−178.6 (3)
C1—N1—C2—C4	−37.6 (4)	C2—C3—C1*c*—C2*c*	163.5 (3)
N1—C1—C1*a*—C2*a*	−20.3 (4)		

24CLPD and 24MOPD were synthesized according to a previously described procedure (Dryanska, 1990). 24CLPD: m.p. 436-437 K (from ethanol/ethylacetate); ^{1}H NMR (80 MHz, $CDCl_3$) δ 6.94 (*s*, 1H, CH=), 7.00-8.00 (*m*, 13H, H_{phenyl}); elemental analysis found/calculated C 70.16/70.04, H 3.78/3.74, N 7.20/7.43%. 24MOPD: m.p. 410-411 K (from ethanol); ^{1}H NMR (80 MHz, $CDCl_3$) δ 3.72, 3.78 (*ss*, 6H, $2CH_3O$), 7.01 (*s*, 1H, CH=), 6.90-7.90 (*m*, 13H, H_{phenyl}); elemental analysis found/calculated C 78.49/78.24, H 5.77/5.47, N 7.90/7.60%.

Data collection: *CAD*-4 (Enraf-Nonius, 1988). Data reduction: *SDP/PDP* (Enraf-Nonius, 1985). Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *SDP/PDP*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *KAPPA* (Maciček, 1992).

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry, along with additional packing diagrams, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71423 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1045]

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Structure of *N*-Acetyl-L-prolylglycinamide

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Abstract

In the crystal, two *N*-acetyl-L-prolylglycinamide molecules form the asymmetric unit and are linked as a dimer by two hydrogen bonds between the N—H groups and amidic O atoms. Each molecule shares six hydrogen bonds with three translated conformers. All the hydrogen bonds lie approximately in planes, which separate the apolar regions. In both independent molecules, the puckering of the proline rings can be described as slightly distorted C_2–C^β-*exo* conformations.

Comment

The structures of small peptides provide information that proves useful in conformational studies on proteins. It appears that short-range interactions are dominant in determining the preferred conformations in polypeptide chains. In particular, the structures of oligopeptides containing prolyl residues have been investigated extensively, owing to the conformational restrictions imposed by the presence of pyrrolidine rings both on the main chain and on residues adjacent to the prolyl residues.

Our recent studies, on the solid-state properties of substituted peptides (*N*-acetylamides), have been concerned with crystallographic determinations (Puliti, Mattia, Barone & Giancola, 1989, 1991; Puliti, Mattia & Lilley, 1992, and references therein) and with the analysis of some thermodynamic parameters connected with phase transitions, the trends of which have been discussed on the basis of crystallographic results (Puliti, Mattia, Barone, Della Gatta & Ferro, 1990; Barone, Giancola, Lilley, Mattia & Puliti, 1992). As a continuation of this program, we present herein the crystal structure of *N*-acetyl-L-prolylglycinamide (NAPGA).

Fig. 1 shows a perspective view of the two (*A* and *B*) NAPGA molecules that form the asymmetric unit. The conformations of the independent molecules are practically equivalent, as the inverted conformations assumed by the two glycinamide moieties (see torsion angles in Table 2) are stereochemically equivalent. The peptide linkages between Pro and Gly residues are, for both molecules, in distorted *trans* forms. The pairs of torsion angles φ, ψ fall in the *FF** (for molecule *A*) and *FF* (for molecule *B*) regions, following the classification of Zimmerman, Pottle, Némethy & Scheraga (1977). In several peptide structures, the glycyl residue occurs in the *F* (or *F**) region, although this area does not correspond to an energy minimum for this single amino acid. In fact, the potential-energy map is quite flat and small changes in the non-bonded atom interactions are sufficient to produce a local minimum (Vasquez, Némethy & Scheraga, 1983).

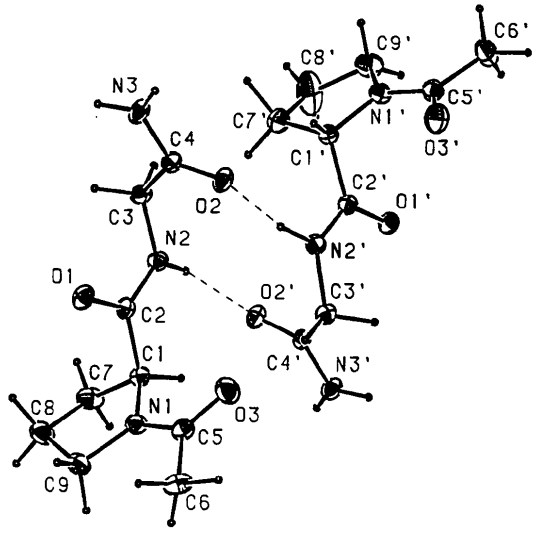

Fig. 1. Perspective drawing of the two NAPGA molecules, which form the asymmetric unit, linked as a dimer by two hydrogen bonds (dashed lines). Unprimed and primed labels refer to molecules *A* and *B*, respectively. Thermal ellipsoids are shown at 30% probability levels.

On average, the intramolecular geometry agrees well with the generally accepted values (Puliti, Mattia & Lilley, 1992) and, in particular, the geometry about N1 and N1′ agrees with the reported values for a peptide group that precedes a *trans* proline (Benedetti, 1982). Except for the values involving C^{γ}-proline, all the differences between the corresponding bond lengths and angles in the two molecules are within experimental error. In molecule *B*, the bond-length values of C7′—C8′ [1.438 (6) Å] and the valency angle C7′—C8′—C9′ [109.3 (4)°], compared with the expected values of 1.52 Å and 105°, can be ascribed to the relatively inaccurate localization of the C8′ atom, which refines with a rather high temperature factor and is strongly anisotropic in a direction almost perpendicular to the average plane of the pyrrolidine ring (see Fig. 1). These features could suggest conformational disorder at C^{γ}, which has been observed in several proline rings (Panneerselvam, Chacko & Veena, 1990) but a careful check of the difference Fourier map, calculated without C8′ contribution, did not show a reliable split position for this atom.

The pyrrolidine ring, which requires a rather modest energy input for deformation, adopts, in both molecules, a C_2–C^{β}-*exo* conformation, distorted towards the C_s–C^{γ}-*endo* form, C^{β} and C^{γ} being displaced in opposite directions with respect to the planes through the remaining ring atoms [C7 0.349 (3), C8 0.174 (3) and C7′ 0.259 (3), C8′ 0.079 (5) Å]. The puckering parameters (Cremer & Pople, 1975) are $q = 0.320$ (4) Å, $\varphi = 84.3$ (7)° and $q = 0.208$ (6) Å, $\varphi = 80.8$ (8)° in molecules *A* and *B*, respectively. These conformations, with torsion angles $\theta = -13.6$ (2) and -10.1 (3)°, are in agreement with the global energy minimum observed, for a proline 'type B' geometry, at $\theta = -10°$ (Venkatachalam, Price & Krimm, 1974). The general trend of the prolyl-ring torsion angles, the smallest of which are θ and χ_4, agrees with the theoretical results, supported by crystallographic evidence, on the conformational flexibility of the proline ring and interdependence between the side-chain and backbone torsion angles (Madison, 1977).

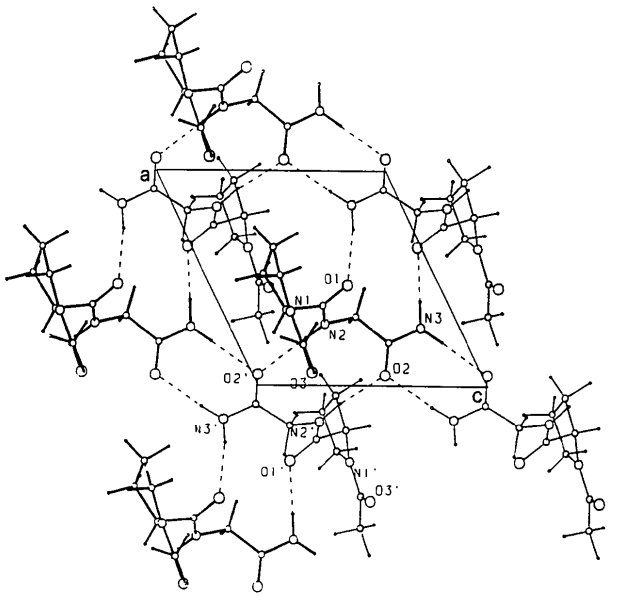

Fig. 2. Crystal packing projected in the *ac* plane: thick and fine lines indicate molecules *A* and *B*, respectively. For clarity, the molecules related by the screw axis have been omitted and only O and N atoms are labelled. Dashed lines indicate hydrogen bonds; the symmetry of the acceptor is reported in Table 3.

The crystal packing is shown in Fig. 2. All the donor groups are engaged in intermolecular hydrogen bonds that connect *A* and *B* molecules so that each NAPGA molecule shares six hydrogen bonds with three translated conformers. The hydrogen-bond patterns involve the same pairs of donor and acceptor atoms in the two molecules. They are linked, by two hydrogen bonds between the N—H groups and amidic O atoms, as in a dimer, whose carbonylglycinamide fragments [from C2 (C2′) to N3 (N3′)] can be related, within 0.2 Å, by a pseudo-inversion centre. Dimers, translated along the *ac* plane, are interconnected by the remaining four hydrogen bonds, which link the amidic N3 and N3′ atoms with the peptidic and amidic O1′ and O2′ or O1 and O2 atoms, respectively. All the hydrogen bonds lie approximately in planes parallel to the *ac* plane and separate apolar regions which contain the prolyl residues at distances >3.9 Å. The short intermolecular contact C3′···O3 = 3.312 (3) Å [H5′···O3 = 2.34 (3) Å and C3′—H5′···O3 = 162 (2)°] can be still considered as a weak C^{α}—H···O hydrogen bond (Taylor & Kennard, 1982; Desiraju, 1991). These electrostatic interactions occur quite frequently in molecules with activated C—H groups and have been seen in several amino acid derivatives (Jeffrey & Maluszynska, 1982). In contrast, for the short contact C9′···O1^{i} = 3.288 (4) Å [symmetry code: (i) $-x$, $-\frac{1}{2}+y$, $1-z$], the distance found for H12′···O1 = 2.52 (5) Å is beyond the accepted value for a C—H···O attractive interaction, for which the H···acceptor distance must be appreciably shorter (at least by 0.3 Å) than the sum of the van der Waals radii (Taylor & Kennard, 1982); in this case the distance is only 0.18 Å shorter.

Experimental

Crystal data

$C_9H_{15}N_3O_3$
M_r = 213.24
Monoclinic
$P2_1$
a = 8.282 (1) Å
b = 17.099 (2) Å
c = 8.367 (1) Å
β = 117.08 (2)°
V = 1055.0 (6) Å^3
Z = 4
D_x = 1.342 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54056 Å
Cell parameters from 24 reflections
θ = 21–27°
μ = 0.813 mm^{-1}
T = 293 K
Prism
0.45 × 0.13 × 0.04 mm
Colourless
Crystal source: slow evaporation from MeOH

Data collection

Enraf–Nonius CAD-4F diffractometer
ω scans
Absorption correction: none
2247 measured reflections
2247 independent reflections
2177 observed reflections [$I > 3.0\sigma(I)$]
θ_{max} = 75°
$h = -10 \rightarrow 9$
$k = 0 \rightarrow 21$
$l = 0 \rightarrow 10$
3 standard reflections
frequency: 240 min
intensity variation: 4%

Refinement

Refinement on F
R = 0.036
wR = 0.038
S = 0.89
2177 reflections
361 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o)+(0.01F_o)^2+1]$ (Killean & Lawrence, 1969)
$(\Delta/\sigma)_{max}$ = 0.35
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: Stout & Jensen (1968)
Extinction coefficient: 7.1 (2) × 10^{-6}
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)

$B_{eq} = \frac{1}{3}\Sigma_i\Sigma_j B_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$. Primed atoms refer to molecule *B*.

	x	y	z	B_{eq}
O1	0.4690 (2)	0.500	0.5984 (2)	3.75 (4)
O2	0.0491 (2)	0.3997 (1)	0.5767 (2)	3.82 (4)
O3	0.0619 (3)	0.5955 (1)	0.2621 (3)	5.09 (5)
N1	0.3455 (3)	0.5996 (1)	0.2942 (3)	3.36 (4)
N2	0.2886 (2)	0.4050 (1)	0.4200 (2)	2.94 (4)
N3	0.2722 (3)	0.3508 (2)	0.8311 (3)	4.37 (5)
C1	0.3536 (3)	0.5153 (2)	0.2789 (3)	3.16 (5)
C2	0.3726 (3)	0.4735 (2)	0.4464 (3)	2.76 (4)

C3	0.3218 (3)	0.3541 (2)	0.5700 (3)	3.31 (5)
C4	0.2018 (3)	0.3718 (2)	0.6599 (3)	3.10 (5)
C5	0.1932 (3)	0.6344 (2)	0.2790 (3)	3.68 (6)
C6	0.1914 (4)	0.7221 (2)	0.2824 (4)	4.59 (7)
C7	0.5237 (4)	0.5028 (2)	0.2544 (3)	4.57 (6)
C8	0.6446 (4)	0.5713 (2)	0.3503 (4)	5.21 (7)
C9	0.5149 (4)	0.6384 (2)	0.3240 (4)	4.34 (6)
O1′	−0.3527 (2)	0.2988 (1)	−0.0179 (2)	3.49 (4)
O2′	0.0529 (2)	0.3763 (1)	0.0221 (2)	3.57 (4)
O3′	−0.5431 (3)	0.2923 (2)	0.2486 (3)	5.35 (5)
N1′	−0.3573 (3)	0.2000 (1)	0.2410 (3)	3.48 (4)
N2′	−0.1692 (2)	0.3837 (1)	0.1909 (2)	3.02 (4)
N3′	−0.1575 (3)	0.4390 (2)	−0.2185 (3)	3.83 (5)
C1′	−0.2095 (3)	0.2555 (2)	0.2898 (3)	3.36 (5)
C2′	−0.2519 (3)	0.3147 (2)	0.1408 (3)	2.82 (4)
C3′	−0.1863 (3)	0.4408 (2)	0.0557 (3)	3.13 (5)
C4′	−0.0877 (3)	0.4146 (2)	−0.0493 (3)	2.84 (5)
C5′	−0.5202 (3)	0.2241 (2)	0.2164 (3)	3.86 (6)
C6′	−0.6722 (4)	0.1653 (3)	0.1469 (5)	5.88 (9)
C7′	−0.0467 (4)	0.2046 (2)	0.3179 (4)	5.23 (8)
C8′	−0.1210 (5)	0.1324 (3)	0.2255 (7)	9.1 (1)
C9′	−0.3079 (4)	0.1221 (2)	0.2058 (4)	5.03 (7)

Table 2. *Geometric parameters* (Å, °)

N1—C1	1.451 (3)	N1′—C1′	1.453 (3)
N1—C5	1.348 (4)	N1′—C5′	1.334 (4)
N1—C9	1.467 (4)	N1′—C9′	1.463 (4)
N2—C2	1.328 (3)	N2′—C2′	1.334 (3)
N2—C3	1.447 (3)	N2′—C3′	1.452 (3)
C1—C2	1.516 (4)	C1′—C2′	1.517 (4)
C1—C7	1.529 (4)	C1′—C7′	1.531 (4)
C3—C4	1.525 (4)	C3′—C4′	1.514 (4)
C7—C8	1.512 (4)	C7′—C8′	1.438 (6)
C8—C9	1.518 (5)	C8′—C9′	1.491 (6)
C1—N1—C5	120.7 (2)	C1′—N1′—C5′	120.3 (2)
C1—N1—C9	112.7 (2)	C1′—N1′—C9′	112.0 (2)
C5—N1—C9	126.6 (2)	C5′—N1′—C9′	127.3 (2)
C2—N2—C3	120.5 (2)	C2′—N2′—C3′	119.8 (2)
N1—C1—C2	112.1 (2)	N1′—C1′—C2′	110.7 (2)
C2—C1—C7	110.1 (2)	C2′—C1′—C7′	111.3 (3)
N2—C2—C1	115.8 (2)	N2′—C2′—C1′	116.0 (2)
N2—C3—C4	113.6 (2)	N2′—C3′—C4′	111.5 (2)
O2—C4—C3	122.1 (2)	O2′—C4′—C3′	121.2 (2)
N3—C4—C3	114.5 (2)	N3′—C4′—C3′	115.9 (2)
O3—C5—N1	120.9 (3)	O3′—C5′—N1′	120.3 (3)
N1—C5—C6	117.3 (3)	N1′—C5′—C6′	117.5 (3)

C5—N1—C1—C2	φ_1	−75.7 (3)
C9—N1—C1—C7	θ	−13.6 (2)
C1—N1—C5—C6	ω'	−175.4 (2)
C1—N1—C9—C8	χ_4	−6.8 (3)
C3—N2—C2—C1	ω	169.6 (2)
C2—N2—C3—C4	φ_2	86.4 (3)
N1—C1—C2—N2	ψ_1	145.2 (2)
N1—C1—C7—C8	χ_1	28.5 (2)
N2—C3—C4—N3	ψ_2	−154.1 (2)
C1—C7—C8—C9	χ_2	−33.1 (3)
C7—C8—C9—N1	χ_3	24.6 (3)
C5′—N1′—C1′—C2′	φ_1	−64.2 (3)
C9′—N1′—C1′—C7′	θ	−10.1 (3)
C1′—N1′—C5′—C6′	ω'	173.6 (2)
C1′—N1′—C9′—C8′	χ_4	−3.1 (3)
C3′—N2′—C2′—C1′	ω	173.4 (2)
C2′—N2′—C3′—C4′	φ_2	−68.8 (3)
N1′—C1′—C2′—N2′	ψ_1	154.4 (2)
N1′—C1′—C7′—C8′	χ_1	19.7 (3)
N2′—C3′—C4′—N3′	ψ_2	149.6 (2)
C1′—C7′—C8′—C9′	χ_2	−22.7 (4)
C7′—C8′—C9′—N1′	χ_3	16.3 (4)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···*A*	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···O2′^{i}	2.15 (3)	3.036 (2)	165 (3)
N2′—H1′···O2^{i}	1.92 (3)	2.907 (2)	169 (3)
N3—H2···O1′^{ii}	1.94 (3)	2.910 (3)	177 (4)
N3—H3···O2′^{iii}	2.02 (4)	2.947 (3)	174 (4)
N3′—H2′···O1^{iv}	2.06 (3)	2.947 (2)	170 (3)
N3′—H3′···O2^{v}	2.09 (4)	2.998 (3)	176 (3)

Symmetry codes: (i) x, y, z; (ii) $1 + x, y, 1 + z$; (iii) $x, y, 1 + z$; (iv) $-1 + x, y, -1 + z$; (v) $x, y, -1 + z$.

The synthesis of *N*-acetyl-L-prolylglycinamide (NAPGA) has been described previously (Milburn, 1984; Lilley, 1988).

The structure was solved by means of the *MULTAN*11/82 package (Main *et al.*, 1982). All calculations were performed using Enraf–Nonius *SDP* software (B. A. Frenz & Associates, Inc., 1985) on a MicroVAX 3100 computer.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71361 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1032]

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