Fig. 3. C—N bond length *versus* torsion angle of the NO$_2$ group. The points are labeled 'molecule-group'. The least-squares line $d = 1.424 + 0.000409\alpha$ is drawn.

order would be a maximum if the NO$_2$ group were coplanar with the ring and a minimum with the NO$_2$ group at 90° from the ring. In a study of the conformations of a large number of nitro-substituted

aromatic rings Holden & Dickenson (1977) found no correlation between C—N distances and torsion angles.

The existence of C—H$\cdots$O hydrogen bonds has been noted by Sutor (1962). Such bonds could exist in the present structure and possible intermolecular hydrogen bonds are listed in Table 2. O$\cdots$H distances less than 2.7 Å are plotted as dotted lines in Fig. 2.

It is unusual, though hardly rare, to find two molecules in the asymmetric unit. There are, however, no close intermolecular contacts which suggest a dimer as the packing unit.

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Structure and Conformation of *cyclo*(-L-Phenylalanyl-L-prolyl-D-alanyl-), Form II of a Synthetic Cyclic Hexapeptide

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Abstract. C$_{34}$H$_{42}$N$_6$O$_6$.5H$_2$O, $M_r = 720.8$, monoclinic, $P2_1$, $a = 13.475$ (1), $b = 9.361$ (2), $c = 15.226$ (1) Å, $\beta = 100.77$ (2)°, $Z = 2$, $V = 1887$ Å^3, $D_x = 1.269$ g cm^{-3}, D_m not measured, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 7.5$ cm^{-1}, $F(000) = 772$, $R = 0.044$, $wR = 0.049$ for 2979 reflections [$I > 2\sigma(I)$], 4232 unique reflections measured. The synthetic cyclic hexapeptide *cyclo*(-L-Phe-L-Pro-D-Ala-)$_2$ crystallizes in

two forms. Form I, crystallized from Me$_2$SO/H$_2$O mixture [$a = 18.730$ (2), $b = 9.239$ (1), $c = 21.544$ (2) Å, $\beta = 95.17$ (2)°, $Z = 4$, $V = 3713$ Å^3], contains two hexapeptide molecules and eight water molecules in the asymmetric unit [Kartha, Bhandary, Kopple, Zhu & Go (1984). *J. Am. Chem. Soc.* **106**, 3844–3850], while form II, crystallized from H$_2$O/ MeOH/NaSCN solution, contains one hexapeptide molecule and five water molecules in the asymmetric unit. The conformation of the hexapeptide molecule

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remains the same even though it is crystallized from two different environments. The hexapeptide has a *two-cis* backbone with *cis* peptide links between L-Phe-L-Pro residues.

Introduction. Recently we reported (Kartha, Bhandary, Kopple, Zhu & Go, 1984) the synthesis and conformational study by X-ray and NMR of the synthetic cyclic hexapeptide *cyclo*(-L-Phe-L-Pro-D-Ala-)$_2$, the crystals of which were grown from an Me$_2$So/H$_2$O mixture. By crystallizing the cyclic peptide in different solvents one might examine the influence of the nature of the solvent on the conformation. Examples of this can be found in the structural studies of *cyclo*-(-L-Leu-L-Tyr-δAva-δAva-) (Karle, 1976; Karle & Flippen-Anderson, 1978), [Phe[4],Val[6]]-antamanide (Karle, 1977; Karle & Duesler, 1977), cycloamanide A (Chiang, Karle & Wieland, 1982; Karle & Chiang, 1984) and valinomycin (Smith *et al.*, 1975; Karle, 1975). In each of these cases the conformation of the peptide remained the same when it was crystallized under different conditions.

In this paper we report the structure of a second form of *cyclo*(-L-Phe-L-Pro-D-Ala-)$_2$ that demonstrates the conformational stability of this hexapeptide. Important features of the conformation are the presence of two *cis* peptide links in both forms, and the absence of transannular hydrogen bonding or the β turns that have been observed in the all-*trans* forms of *cyclo*-(XXX-L-Pro-D-YYY-)$_2$ peptides (Brown & Yang, 1979; Brown & Teller, 1976; Kostansek, Lipscomb & Thiessen, 1979).

Experimental. Crystal $0.8 \times 0.5 \times 0.3$ mm sealed in a capillary. Enraf–Nonius CAD-4 automated diffractometer, Ni-filtered Cu $K\alpha$, ω–2θ scans and integrated counts with $\theta < 77°$, $0 < h < 17$, $0 < k < 11$, $-19 < l < 19$, 4232 independent reflections, 2979 with $I > 2\sigma(I)$. Lattice parameters measured using 25 reflections ($18.9° < \theta < 46.1°$). Three reflections (700, 227, 0$\bar{4}$1) serving as monitors were measured at intervals of 2 h; indicated no crystal deterioration. Lp corrections, empirical absorption corrections (North, Phillips & Mathews, 1968). Direct methods, *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least-squares refinement for 51 non-hydrogen and 42 hydrogen atoms. Anisotropic thermal parameters for non-hydrogen atoms. Hydrogen atoms located from difference Fourier map, refined with isotropic thermal parameters. $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1/\sigma(F^2)$, $(\Delta/\sigma)_{max} = 0.51$, $\Delta\rho_{max} = 0.23$ e Å^{-3}, $\Delta\rho_{min} = -0.21$ e Å^{-3}. Final $R = 0.044$, $wR = 0.049$ for 2979 reflections ($R = 0.074$ for 4232 reflections), $S = 2.891$. SDP package and local programs. Extinction coefficient 3.242×10^{-6}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The final atomic parameters are given in Table 1.* Table 2 lists bond lengths, angles and torsional angles. The hexapeptide has two *cis* peptide links between the L-Phe and L-Pro residues with values of -19.8 and $-11.6°$. The remaining four peptide links are *trans* with the non-planarity parameter ω varying by 7° or less. Three molecules of this peptide have now been observed, this one, in the form II crystal, and two in the form I crystal. All have peptide backbones that are similar and approximately C_2 symmetric, so that one can examine the distribution of conformations of

Table 1. *Positional parameters and their e.s.d.'s*

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $\frac{4}{3}[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + (ab\cos\gamma)\beta(1,2) + (ac\cos\beta)\beta(1,3) + (bc\cos\alpha)\beta(2,3)]$.

	x	y	z	$B_{eq}(\text{Å}^2)$
N(1)	0.0814 (3)	0.428	0.3852 (2)	3.84 (8)
C(1α)	0.1148 (3)	0.3622 (6)	0.3077 (3)	3.6 (1)
C(1β)	0.0305 (4)	0.2661 (7)	0.2542 (3)	4.9 (1)
C(1γ)	0.0573 (4)	0.2046 (6)	0.1713 (3)	4.6 (1)
C(1δ1)	0.0253 (5)	0.2646 (8)	0.0895 (4)	6.8 (2)
C(1ϵ1)	0.0532 (5)	0.213 (1)	0.0125 (4)	9.6 (2)
C(1ζ)	0.1154 (6)	0.099 (1)	0.0220 (5)	12.7 (3)
C(1ϵ2)	0.1483 (6)	0.036 (1)	0.1005 (5)	10.8 (2)
C(1δ2)	0.1184 (5)	0.0872 (7)	0.1745 (4)	7.2 (2)
C(1')	0.1391 (3)	0.4789 (5)	0.2473 (3)	3.6 (1)
O(1)	0.0784 (2)	0.5795 (4)	0.2257 (2)	4.87 (8)
N(2)	0.2198 (3)	0.4668 (4)	0.2099 (2)	3.53 (8)
C(2α)	0.3074 (3)	0.3736 (5)	0.2400 (3)	3.24 (9)
C(2β)	0.3705 (4)	0.3892 (7)	0.1654 (3)	4.9 (1)
C(2γ)	0.3051 (4)	0.4671 (7)	0.0911 (3)	5.3 (1)
C(2δ)	0.2359 (4)	0.5576 (6)	0.1353 (3)	4.7 (1)
C(2')	0.3670 (3)	0.4275 (5)	0.3301 (3)	3.12 (9)
O(2)	0.3399 (2)	0.5290 (4)	0.3703 (2)	4.59 (8)
N(3)	0.4513 (3)	0.3525 (4)	0.3604 (2)	3.19 (8)
C(3α)	0.5146 (3)	0.3846 (5)	0.4471 (2)	3.11 (9)
C(3β)	0.6112 (3)	0.2956 (6)	0.4586 (3)	4.1 (1)
C(3')	0.4568 (3)	0.3495 (5)	0.5217 (3)	3.33 (9)
O(3)	0.3967 (2)	0.2472 (4)	0.5126 (2)	4.84 (8)
N(4)	0.4786 (3)	0.4257 (4)	0.5960 (2)	3.37 (8)
C(4α)	0.4359 (3)	0.3874 (5)	0.6735 (3)	3.37 (9)
C(4β)	0.5080 (4)	0.2822 (6)	0.7324 (3)	4.5 (1)
C(4γ)	0.4713 (3)	0.2376 (6)	0.8161 (3)	3.9 (1)
C(4δ1)	0.5058 (4)	0.3065 (6)	0.8967 (3)	4.6 (1)
C(4ϵ1)	0.4709 (4)	0.2686 (7)	0.9728 (3)	5.5 (1)
C(4ζ)	0.4016 (4)	0.1608 (7)	0.9713 (3)	6.0 (1)
C(4ϵ2)	0.3672 (4)	0.0900 (7)	0.8923 (4)	6.1 (2)
C(4δ2)	0.4021 (4)	0.1281 (6)	0.8146 (3)	4.8 (1)
C(4')	0.4255 (3)	0.5194 (5)	0.7294 (3)	3.17 (9)
O(4)	0.4960 (2)	0.6059 (4)	0.7457 (2)	4.24 (7)
N(5)	0.3429 (3)	0.5341 (4)	0.7658 (2)	3.32 (8)
C(5α)	0.2497 (3)	0.4486 (5)	0.7457 (3)	3.3 (1)
C(5β)	0.2007 (4)	0.4801 (6)	0.8276 (3)	4.5 (1)
C(5γ)	0.2331 (4)	0.6302 (6)	0.8542 (3)	4.5 (1)
C(5δ)	0.3395 (4)	0.6450 (6)	0.8340 (3)	4.3 (1)
C(5')	0.1838 (3)	0.5002 (5)	0.6587 (3)	3.4 (1)
O(5)	0.1931 (3)	0.6192 (4)	0.6286 (2)	5.95 (9)
N(6)	0.1157 (3)	0.4050 (4)	0.6208 (2)	3.61 (8)
C(6α)	0.0470 (3)	0.4316 (6)	0.5367 (3)	3.8 (1)
C(6β)	−0.0597 (4)	0.3787 (7)	0.5416 (3)	5.0 (1)
C(6')	0.0851 (3)	0.3564 (5)	0.4610 (3)	3.6 (1)
O(6)	0.1158 (3)	0.2311 (4)	0.4702 (2)	5.67 (9)
OW(1)	0.2140 (3)	0.8995 (5)	0.6985 (2)	5.95 (9)
OW(2)	0.2326 (3)	0.7855 (5)	0.3194 (3)	6.8 (1)
OW(3)	0.9006 (3)	0.6468 (5)	0.2789 (3)	7.1 (1)
OW(4)	0.7362 (3)	0.6063 (4)	0.4084 (3)	6.15 (9)
OW(5)	0.6867 (3)	0.5631 (4)	0.6353 (2)	5.64 (9)

six C_2 halves. The greatest deviations from C_2 symmetry occur in the present case: there are φ values of 66·6 and 101·1° for D-Ala(3) and D-Ala(6) respectively, and values ψ for Pro(2) and Pro(5) of −176·9 and 160·1°. These different angles correspond to a difference, in the two halves of the molecule, in rotation of the Pro—Ala CONH plane relative to the average peptide ring plane. The two conformations at Pro-D-Ala found in form II are also each represented, together with an intermediate set of Pro-D-Ala angles, in one of the two molecules of form I. It is likely that the Pro-D-Ala part of the molecule is one in which the stable conformation lies in a broad energy minimum. The conformation is illustrated in Fig. 1.

Table 2. *Bond lengths* (Å; *average e.s.d.* 0·004 Å), *bond angles* (°; *average e.s.d.* 0·3°) *and torsion angles* (°; *average e.s.d.* 0·6°) *for the title compound*

	Phe 1	Pro 2	D-Ala 3	Phe 4	Pro 5	D-Ala 6
N(i)—C(ia)	1·472	1·471	1·462	1·452	1·472	1·454
C(ia)—C(i)'	1·504	1·539	1·528	1·522	1·529	1·519
C(i)'—O(i)	1·250	1·222	1·244	1·237	1·220	1·243
C(i)'—N(i + 1)	1·323	1·342	1·323	1·340	1·331	1·325
C(ia)—C(iβ)	1·555	1·548	1·528	1·546	1·546	1·535
C(iβ)—C(iγ)	1·491	1·488		1·509	1·504	
C(iγ)—C(iδ)		1·508			1·527	
C(iδ)—N(i)		1·467			1·475	
C(iγ)—C(iδ1)	1·361			1·388		
C(iδ1)—C(iε1)	1·384			1·376		
C(iε1)—C(iδ)	1·351			1·372		
C(iζ)—C(iε2)	1·332			1·376		
C(iε2)—C(iδ2)	1·354			1·398		
C(iδ2)—C(iγ)	1·369			1·383		
C(i)'−1−N(i)—C(ia)	121·5	126·4	121·1	120·3	126·9	122·7
C(ia)—N(i)—C(iδ)		111·0			113·1	
C(i)'−1−N(i)—C(iδ)		122·4			120·1	
C(i)'—C(ia)—N(i)	108·8	109·7	109·6	110·2	110·1	109·8
N(i)—C(ia)—C(iβ)	110·9	103·5	109·4	109·1	101·8	110·5
C(iβ)—C(ia)—C(i)'	108·7	110·9	109·9	107·9	111·8	109·9
C(ia)—C(i)'—O(i)	120·1	123·1	119·5	119·7	122·0	120·3
C(ia)—C(i)'—N(i + 1)	119·4	113·7	117·2	119·0	114·0	117·7
N(i + 1)—C(i)'—O(i)	120·1	123·2	123·1	121·1	124·0	121·9
C(ia)—C(iβ)—C(iγ)		106·0			104·4	
C(iβ)—C(iγ)—C(iδ)		105·3			105·5	
C(iγ)—C(iδ)—N(i)		102·3			103·4	
C(ia)—C(iβ)—C(iγ)	113·5			113·4		
C(iβ)—C(iγ)—C(iδ1)	122·0			120·7		
C(iγ)—C(iδ1)—C(iε1)	122·8			120·9		
C(iδ1)—C(iε1)—C(iζ)	116·6			121·0		
C(iε1)—C(iζ)—C(iε2)	122·9			119·0		
C(iζ)—C(iε2)—C(iδ2)	119·2			120·4		
C(iε2)—C(iδ2)—C(iγ)	121·9			120·5		
C(iδ2)—C(iγ)—C(iδ1)	116·7			118·2		
C(iβ)—C(iγ)—C(iδ2)	121·3			121·2		

Angle $i=$		Phe 1	Pro 2	D-Ala 3	Phe 4	Pro 5	D-Ala 6
C(i)'−1−N(i)—C(ia)—C(i)'	φ	−158·8	−68·5	66·6	−151·6	−79·4	101·1
N(i)—C(ia)—C(i)'—N(i + 1)	ψ	138·9	−176·9	−150·3	140·0	160·1	−137·0
C(ia)—C(i)'—N(i+1)—C(i+1a)	ω	−19·8	−177·0	−172·5	−11·6	−178·6	−178·8
C(iδ)—N(i)—C(ia)—C(iβ)	χ^0		−11·9			−17·8	
N(i)—C(ia)—C(iβ)—C(iγ)	χ^1		−10·5			31·5	
C(ia)—C(iβ)—C(iγ)—C(iδ)	χ^2		28·2			−34·4	
C(iβ)—C(iγ)—C(iδ)—N(i)	χ^3		−34·6			23·1	
C(iγ)—C(iδ)—N(i)—C(ia)	χ^4		29·0			−2·8	
N(i)—C(ia)—C(iβ)—C(iγ)	χ^1	176·1			178·9		
C(ia)—C(iβ)—C(iγ)—C(iδ1)	χ^2	−97·8			−96·2		
C(iδ2)—C(iγ)—C(iδ1)—C(iε1)	χ^3	−0·9			−1·1		
C(iγ)—C(iδ1)—C(iε1)—C(iζ)	χ^4	−0·4			0·6		
C(iδ1)—C(iε1)—C(iζ)—C(iε2)	χ^5	0·8			0·1		
C(iε1)—C(iζ)—C(iε2)—C(iδ2)	χ^6	0·3			−0·3		
C(iζ)—C(iε2)—C(iδ2)—C(iγ)	χ^7	−1·8			−0·2		
C(iε2)—C(iδ2)—C(iγ)—C(iδ1)	χ^8	2·0			0·9		

The pseudorotational parameters (Altona & Sundaralingam, 1972) calculated for the proline rings in both forms show varying conformations. In form I the two proline rings of molecule A have conformations that can be described (Hasnoot, DeLeeuw, Deleeuw & Altona, 1981) as $^\beta E$[Pro(2A): $P=35\cdot5°$, $\tau_m=166\cdot0°$] and a conformation between $^\beta E$ and $^\beta_\gamma T$[Pro(5A): $P = 35\cdot8$, $\tau_m = 173\cdot2$]. In molecule B of form I the two prolines have $_\alpha E$[Pro(2B): $P = 20\cdot4$, $\tau_m = 128\cdot0$] and $^\gamma_\beta T$[Pro(5B): $P = 33\cdot7$ and $\tau_m = 31\cdot6$]. In form II the proline residue Pro(2) has a $^\gamma_\beta T(P = 35\cdot1$, $\tau_m = 37\cdot2)$ while Pro(5) has a $^\beta E(P = 34\cdot8$, $\tau_m = 167\cdot2)$ conformation. Hasnoot *et al.* (1981) have also noted that in cyclic tri-, penta-, and decapeptides the ring strain dictates an upward deviation from planarity about N $(\chi_0 - \varphi \simeq 70 \pm 6°)$. In the present analysis of the proline rings in the two forms of the cyclic hexapeptide the average $\chi_0 - \varphi$ value is 57 ±6° which is close to 60°.

There are five water molecules per molecule of the hexapeptide in form II while there are four molecules per peptide molecule in form I. The crystal packing of the molecule in form II as viewed down the b axis and the hydrogen bonding are shown in Fig. 2. Five of the six carbonyl oxygens form good hydrogen bonds [O···O distances between 2·684 (5) and 2·994 (5) Å] with the water molecules. One of the phenylalanine carbonyl oxygens [O(4)] is involved in an intermolecular hydrogen bond with the D-alanine amide N(3) [O···N distance of 2·979 (5) Å] of a symmetry-related molecule, thus forming a dimer as in the crystal structure of form I. Two other amides, N(4) and N(6), have contacts of 3·041 (5) and 2·888 (6) Å with OW(5) and OW(3) respectively. The water molecule OW(1) forms hydrogen bonds with OW(3) and OW(4) [O···O distances of 2·839 (6) and 2·694 (6) Å respectively] while OW(2) has a contact distance of 2·852 (6) Å with OW(5).
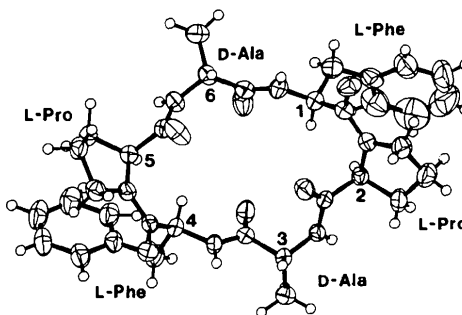
Fig. 1. *ORTEP* drawing (Johnson, 1965) showing the conformation of the hexapeptide. The α-carbons are numbered.
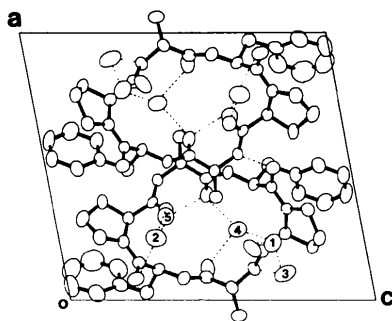
a

Fig. 2. Crystal packing as viewed down the *b* axis. The water oxygens are numbered; broken lines represent hydrogen bonding.

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Structure of Bis(*p*-nitrophenyl) Diselenide at 187 K

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Abstract. $C_{12}H_8N_2O_4Se_2$, $M_r = 402.1$, monoclinic, $C2/c$, $a = 7.2771$ (12), $b = 14.142$ (3), $c = 12.873$ (3) Å, $\beta = 90.51$ (2)°, $V = 1324.7$ Å^3, $Z = 4$, $D_x = 2.02$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.70930$ Å, $\mu = 55.37$ cm^{-1}, $F(000) = 776$, $T = 187$ (1) K, $R = 0.023$ for 1104 observed reflections. The molecule has a twofold crystallographic symmetry axis passing through the Se—Se bond [2.3018 (8) Å]. The phenyl group is asymmetrically coordinated with Se—C—C angles of 123.7 (2) and 115.6 (2)°, and the phenyl ring plane passes 0.105 (5) Å from the Se atom [Se—C 1.920 (3) Å].

Introduction. The structure of the title compound was determined at reduced temperature, to provide high-precision bond lengths and angles prior to attempts to study the anomeric effect in selenium compounds.

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Experimental. A single crystal measuring $0.020 \times 0.021 \times 0.014$ cm was mounted within a capillary. The crystal was pale yellow in color and showed sharp extinction in polarized light. Reduced temperature was obtained using a modified Nonius Universal Low Temperature apparatus. After cooling from room temperature over a period of 4 h, low temperature was maintained until the end of data collection to avoid stressing the crystal more than necessary. Cell dimensions were determined by least-squares fit of 25 reflections ($14.4 < \theta < 22.4°$) using Mo $K\alpha$ radiation with graphite monochromator. Intensity data were collected on an Enraf–Nonius CAD-4F diffractometer using ω–2θ scans. 1526 independent reflections measured with 1104 considered observed with $[I/\sigma(I)] > 2.5$; $h\ 0\rightarrow9$, $k\ 0\rightarrow18$, $l\ -16\rightarrow16$; $2\theta_{max} = 55°$. Two reflections were used as intensity standards which were measured every hour. Variations in intensity of these standards were less than ±2% (3σ) and