Table 4. *Atom deviations* (Å) *from least-squares planes*

	(I*A*)	(I*B*)	(II)*
N(1)	−0·064	−0·125	0·057
O(15)	−0·026	−0·021	−0·011
O(31)	0·120	0·135	0·008
C(2)	0·016	0·009	0·017
C(3)	0·179	0·140	0·002
C(7)	−0·188	−0·163	−0·033
C(8)	−0·036	−0·017	0·003
R.m.s deviation	0·112	0·107	0·026

* The atoms of the reference structure have been renumbered to correspond to (I).

Table 5. *Torsion angles* (°)

	(I*A*)	(I*B*)
C(8)–N(1)–C(9)–C(10)	35·5	37·0
N(5)–C(4)–C(16)–O(19)	15·5	−11·1
N(5)–C(4)–C(16)–O(17)	−167·2	170·8
N(5)–C(4)–C(20)–C(21)	−61·0	−46·6
N(5)–C(6)–C(26)–C(27)	21·5	−147·6
N(5)–C(6)–C(26)–O(30)	−166·6	34·2

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The Structure of 1-Acetylprolinamide

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Abstract. $C_7H_{12}N_2O_2$, M_r = 156·19, orthorhombic, $P2_12_12_1$, $a = 6·680$ (9), $b = 7·685$ (8), $c = 15·627$ (14) Å, $V = 802·2$ (6) Å^3, $Z = 4$, $D_m = 1·315$, $D_c = 1·294$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ(Mo $K\alpha$) = 0·105 mm^{-1}, F(000) = 336. 1410 observed reflections gave a final R = 0·046. Two strong intermolecular hydrogen bonds involving the H atoms of the amide group determine the crystal structure.

Introduction. Crystals of $C_7H_{12}N_2O_2$ (NACPRO) were obtained by slow evaporation of a purified specimen in acetone. They are transparent and colourless. Precise lattice parameters were obtained by least-squares fitting of the powder pattern.

For the X-ray investigation a large single crystal was ground to a sphere of 0·3 mm radius. Intensities were measured on an automatic four-circle diffractometer, using graphite-monochromated Mo $K\alpha$ radiation and the $\omega/2\theta$ scan mode. Two reflexions were monitored periodically during the data-collection process. Subsequent analysis of these reflexions indicated no crystal decomposition during the time required to collect the intensities. 3724 reflexions were collected in the range $1·3 < \theta < 46°$ of which 1410 were considered as observed according to the criterion $I > 2\sigma(I)$ and used in the calculations.

The solution of this structure was quite difficult. First attempts using an older version of *MULTAN* (Main, Woolfson, Declercq & Germain, 1974) and 2000 $\sum_2$ relationships were without success. This was probably due to the weak representation of reflexions with odd k and to the small number of $\sum_2$ relationships. The structure was finally solved with a newer version of

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MULTAN (Main, Lessinger, Woolfson, Germain & Declercq, 1977) using the 200 strongest E values (calculated using the Debye curve) with $\sin\theta/\lambda < 0{\cdot}6$ Å^{-1} and $E \geq 1{\cdot}229$ and applying 3500 $\sum_2$ relationships. With the reflexions 120 (90°), 0,2,11 (360°) and 415 (45°) as origin- and enantiomorph-defining reflexions and four other reflexions a total of 56 solutions were calculated. The three sets with the highest combined figure of merit gave the correction solution. The corresponding E map showed all the non-hydrogen atoms of the structure as the most prominent peaks.

Structure factors based on these coordinates and assuming an overall temperature factor of 3·5 Å^2 gave an R index of 0·262. The f curves for neutral C, O, N and H (Hanson, Herman, Lea & Skillman, 1964) were used. After four cycles of isotropic full-matrix least-squares refinement for the 1410 observed reflexions and four more cycles with anisotropic temperature factors, the conventional R value for the observed reflexions was 0·080. A difference synthesis calculated with those observed reflexions within $\sin\theta/\lambda < 0{\cdot}5$ Å^{-1} showed all the H atoms as the highest peaks of the map with electron densities ranging from 0·26 to 0·51 e Å^{-3}.

An appropriate weighting scheme for the refinement was $w = w_1 w_2$ where $w_1 = 1/\sigma_1^2$ and $w_2 = 1/\sigma_2^2$ with $\sigma_1 = a + b|F_o|$ and $\sigma_2^2 = c + d(\sin\theta/\lambda) + e(\sin\theta/\lambda)^2$ (coefficients given in Table 1), calculated by the program *PESOS* (Martínez-Ripoll & Cano, 1975). After six cycles of weighted least-squares refinement assuming isotropic temperature factors for the H atoms, the final unweighted and weighted disagreement indices were $R = 0{\cdot}046$ and $R_w = 0{\cdot}047$, where $R_w = (\sum w\Delta^2/\sum w|F_o|^2)^{1/2}$. A final difference synthesis showed no significant electron density. The fractional coordinates are given in Table 2.*

Discussion. Fig. 1 shows the shape of the molecule as it exists in the crystal. The bond lengths and angles are given in Table 3. Those in the proline ring are comparable with those given by Kartha, Ashida & Kakudo (1974) and by Kartha & Ambady (1975). The 1-acetyl group has bond lengths and angles comparable with those found by Lecomte, Aubry, Protas, Boussard & Marraud (1974*a*,*b*).

An examination of the torsion angles (given in Table 4) shows that the conformation of the molecule in the crystal is *endo* (Balasubramanian, Lakshminarayanan, Sabesan, Tegoni, Venkatesan & Ramachandran, 1971).

Due to this conformation, atom C(3) is placed 0·79 Å above the plane through atoms N(1), C(1), C(2). C(1) and the atoms of the amide group lie in a plane with a maximum deviation of 0·007 Å. The angle between this plane and a plane through all the atoms of the proline ring is 79°. This illustrates that the amide group is orientated halfway between *cis* and *trans*.

Table 1. *Coefficients of the weighting scheme*

	a	b
$0 \leq F_o < 0{\cdot}57$	2·46	−3·83
$0{\cdot}57 < F_o < 1{\cdot}92$	0·10	0·33
$1{\cdot}92 < F_o < 3{\cdot}16$	1·27	−0·34
$3{\cdot}16 < F_o < 4{\cdot}39$	0·20	0·003
$4{\cdot}39 < F_o \leq 77{\cdot}20$	0·07	0·03

	c	d	e
$0 \leq \sin\theta/\lambda < 0{\cdot}773$	7·22	−21·44	17·30
$0{\cdot}773 < \sin\theta/\lambda < 0{\cdot}840$	0·75	0·17	0
$0{\cdot}840 < \sin\theta/\lambda < 0{\cdot}897$	−1·86	3·24	0
$0{\cdot}897 < \sin\theta/\lambda \leq 1{\cdot}000$	0·77	0·25	0

Table 2. *Fractional coordinates of NACPRO* ($\times 10^5$; *for* H, $\times 10^3$)

	x	y	z
N(1)	18910 (20)	−57 (18)	3074 (8)
C(1)	11696 (22)	−5837 (20)	11452 (9)
C(2)	28441 (32)	−18013 (26)	14536 (13)
C(3)	47294 (30)	−10054 (28)	10604 (15)
C(4)	40370 (25)	−4231 (27)	1750 (12)
C(5)	7649 (23)	8875 (20)	17800 (9)
N(2)	14729 (26)	24651 (20)	16086 (9)
O(1)	−1781 (30)	5452 (19)	24379 (9)
C(6)	6177 (23)	6037 (22)	−2881 (9)
O(2)	−11766 (19)	8647 (22)	−1224 (8)
C(7)	14741 (36)	9762 (33)	−11596 (11)
H(1)	−3 (4)	−118 (3)	111 (1)
H(2)	268 (4)	−296 (4)	121 (2)
H(3)	286 (4)	−201 (4)	202 (2)
H(4)	521 (4)	9 (3)	139 (1)
H(5)	593 (6)	−179 (4)	101 (2)
H(6)	468 (5)	58 (4)	−3 (1)
H(7)	415 (6)	−131 (5)	−34 (2)
H(8)	250 (7)	27 (6)	−133 (2)
H(9)	67 (8)	106 (7)	−151 (3)
H(10)	228 (7)	202 (6)	−115 (2)
H(11)	124 (4)	321 (4)	188 (2)
H(12)	200 (4)	265 (3)	113 (1)

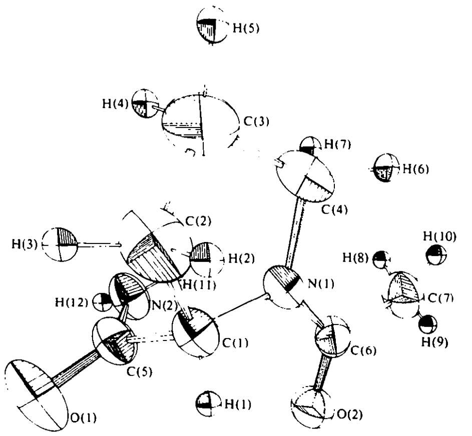

Fig. 1. Molecule of $C_7H_{12}N_2O_2$ (the thermal ellipsoids have been scaled to the 50% probability level).

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33935 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

N(1)–C(1)	1·464 (2)	C(2)–H(2)	0·97 (3)
N(1)–C(4)	1·484 (2)	C(2)–H(3)	0·91 (3)
N(1)–C(6)	1·345 (2)	C(3)–H(4)	1·05 (3)
C(1)–C(2)	1·536 (3)	C(3)–H(5)	1·01 (4)
C(1)–C(5)	1·528 (2)	C(4)–H(6)	0·95 (3)
C(2)–C(3)	1·529 (3)	C(4)–H(7)	1·06 (4)
C(3)–C(4)	1·526 (3)	C(7)–H(8)	0·91 (5)
C(5)–O(1)	1·234 (2)	C(7)–H(9)	0·78 (6)
C(5)–N(2)	1·329 (2)	C(7)–H(10)	0·97 (5)
C(6)–C(7)	1·505 (3)	N(2)–H(11)	0·74 (3)
C(6)–O(2)	1·243 (2)	N(2)–H(12)	0·83 (3)
C(1)–H(1)	0·93 (3)		

C(1)–N(1)–C(4)	112·2 (0·1)	C(3)–C(2)–H(3)	117·2 (2·0)
C(1)–N(1)–C(6)	121·1 (0·1)	H(2)–C(2)–H(3)	102·8 (2·8)
C(4)–N(1)–C(6)	126·2 (0·1)	C(2)–C(3)–H(4)	112·1 (1·7)
N(1)–C(1)–C(2)	103·0 (0·1)	C(4)–C(3)–H(4)	108·3 (1·6)
N(1)–C(1)–C(5)	114·5 (0·1)	C(2)–C(3)–H(5)	116·3 (2·2)
C(2)–C(1)–C(5)	112·1 (0·1)	C(4)–C(3)–H(5)	111·0 (2·1)
C(1)–C(2)–C(3)	103·3 (0·2)	H(4)–C(3)–H(5)	105·7 (2·8)
C(2)–C(3)–C(4)	103·4 (0·2)	N(1)–C(4)–H(6)	108·4 (2·1)
C(3)–C(4)–N(1)	103·3 (0·2)	C(3)–C(4)–H(6)	114·8 (1·9)
C(1)–C(5)–O(1)	118·3 (0·2)	N(1)–C(4)–H(7)	108·4 (2·3)
C(1)–C(5)–N(2)	118·8 (0·1)	C(3)–C(4)–H(7)	118·7 (2·3)
O(1)–C(5)–N(2)	123·0 (0·2)	H(6)–C(4)–H(7)	103·0 (3·0)
N(1)–C(6)–C(7)	116·9 (0·2)	C(6)–C(7)–H(8)	116·0 (3·0)
N(1)–C(6)–O(2)	121·5 (0·2)	C(6)–C(7)–H(9)	114·1 (4·0)
C(7)–C(6)–O(2)	121·6 (0·2)	C(6)–C(7)–H(10)	111·0 (2·8)
N(1)–C(1)–H(1)	112·7 (1·6)	H(8)–C(7)–H(9)	110·8 (5·0)
C(2)–C(1)–H(1)	110·1 (1·7)	H(8)–C(7)–H(10)	94·2 (4·3)
C(5)–C(1)–H(1)	104·7 (1·6)	H(9)–C(7)–H(10)	108·9 (5·0)
C(1)–C(2)–H(2)	110·8 (1·9)	C(5)–N(2)–H(11)	121·3 (2·5)
C(3)–C(2)–H(2)	107·5 (1·9)	C(5)–N(2)–H(12)	119·6 (1·9)
C(1)–C(2)–H(3)	115·3 (2·0)	H(11)–N(2)–H(12)	118·1 (3·1)

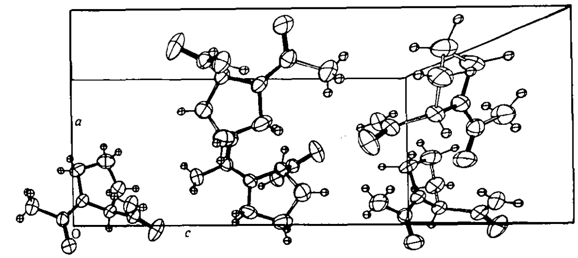

Fig. 2. Unit cell of NACPRO viewed along the *b* axis (the hydrogen bonds are shown with dotted lines).

The crystal structure of NACPRO is shown in Fig. 2. It is determined by two strong intermolecular hydrogen bonds formed by the H atoms of the amide group and the O atoms of neighbouring amide and acetyl groups. The distances are H(11)···O(1') 2·198 (33), N(2)···O(1') 2·928 (2) Å and H(12)··· O(2'') 2·298 (29), N(2)···O(2'') 3·083 (2) Å. The corresponding bond angles are 170·87 (1·88) and 157·70 (3·12)°. In an attempt to form a linear bond, atom H(11) bonds very strongly to N(2) (0·74 Å). The two hydrogen bonds are also the reason for the energetically unfavourable position of the amide group halfway between *cis* and *trans*.

Most of the computations required in this analysis were performed with the XRAY 70 system (Stewart, Kundell & Baldwin, 1970) on the 1106 Univac computer of the Rechenzentrum der Universität Freiburg. This work was supported by a grant from the Deutsche Forschungsgemeinschaft. The authors thank Professor Dr H. Faulstich for a sample of the substance. We wish to thank the Wissenschaftliche Gesellschaft zu Freiburg for a travel grant to one of us (UD) and Professor M. M. Woolfson for his help during our stay at the University of York.

Table 4. *Important torsional angles in the molecule* (°)

The convention used for the labelled angles is that of Balasubramanian *et al.* (1971).

N(1)–C(1)–C(2)–C(3)	32·0 (0·2) χ^1
C(1)–C(2)–C(3)–C(4)	−38·8 (0·2) χ^2
C(2)–C(3)–C(4)–N(1)	30·1 (0·2) χ^3
C(3)–C(4)–N(1)–C(1)	−10·3 (0·2) χ^4
C(4)–N(1)–C(1)–C(2)	−13·6 (0·2) θ
C(5)–C(1)–C(2)–C(3)	−91·5 (0·1) θ'
C(4)–N(1)–C(1)–C(5)	108·3 (0·2) θ''
C(6)–N(1)–C(4)–C(3)	178·6 (0·1) θ'''
C(6)–N(1)–C(1)–C(2)	158·0 (0·2) θ''''
C(6)–N(1)–C(1)–C(5)	−80·1 (0·2) φ
N(1)–C(1)–C(5)–N(2)	−14·2 (0·2) ψ
N(1)–C(1)–C(5)–O(1)	167·1 (0·2)
C(5)–C(1)–C(2)–C(3)	−91·5 (0·2)
C(1)–N(1)–C(6)–C(7)	−173·4 (0·1)
C(1)–N(1)–C(6)–O(2)	7·4 (0·2)
C(2)–C(1)–C(5)–N(2)	102·7 (0·2)
C(2)–C(1)–C(5)–O(1)	−76·0 (0·2)
O(1)–C(5)–N(2)–H(11)	−5·5 (2·9)
O(1)–C(5)–N(2)–H(12)	−174·1 (2·4)

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