

Studies in molecular structure, symmetry and conformation
VI. Crystal and molecular structure of 1-aminocyclopentane carboxylic acid monohydrate

M. MALLIKARJUNAN AND K. K. CHACKO

*Centre of Advanced Study in Physics,
University of Madras, Madras-25, India*

AND

R. ZAND

*Biophysics Research Division, University of Michigan,
Ann Arbor, U.S.A.*

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Abstract

1-aminocyclopentane carboxylic acid monohydrate is monoclinic; space group $P2_1/c$, $a = 11.24$, $b = 6.27$, $c = 11.22 \text{ \AA}$ and $\beta = 97.6^\circ$. The crystal structure was solved by the symbolic addition method and refined to an R factor of 12.1 %. The cyclopentane ring is disordered; one of the carbon atoms exists in two alternative sites, leading to two possible conformations both of which are of the envelope type.

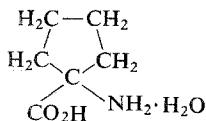
Introduction

Crystal structure analyses of amino cycloalkane carboxylic acid derivatives have been carried out in this laboratory, and some have been reported already (Chandrasekharan *et al.* 1968; Srikrishnan *et al.* 1971 and Chacko *et al.* 1971*a,b*).

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Their molecular structures are of interest from both the point of view of the conformational features of the cycloalkane rings and their nature as amino acids. The hydrobromide derivative of 1-amino cyclopentane carboxylic acid has been reported (Chandrasekharan *et al.* 1968). In this paper we present the structure of 1-aminocyclopentane carboxylic acid monohydrate, the structural formula of which is given below:



Experimental

Good needle-shaped crystals were available for data collection. The unit cell dimensions were determined from precession photographs, and the relevant crystal data are given below:

Molecular formula	C ₆ H ₁₁ O ₂ N·H ₂ O
<i>FW</i>	147
<i>a</i>	11.24(2) Å
<i>b</i>	6.27(1)
<i>c</i>	11.22(2)
β	97.60(5) $^\circ$
<i>V_c</i>	783.9 Å ³
<i>D_m</i>	1.23 g cm ⁻³
<i>D_c</i>	1.25
<i>Z</i>	4
Space group	<i>P</i> 2 ₁ / <i>c</i>
<i>F</i> (000)	320
μ (Cu <i>K</i> α)	9 cm ⁻¹

A crystal of dimensions 0.03, 0.03, 0.05 cm was chosen for collection of intensity data hkl ($k = 0-5$) by the equi-inclination Weissenberg method. The intensities of the reflexions were measured visually by comparison with a standard set of spots recorded with the same crystal. The data were corrected for Lorentz, polarization and spot shape (Phillips, 1962) factors. A total of 1047 reflexions were collected and placed on absolute scale by Wilson's method.

Structure determination and refinement

The structure was solved by the symbolic addition method of Karle & Karle (1963). A total of 165 reflexions with $|E| > 1.3$ was sorted in decreasing order and divided into two groups: (a) those with $E > 1.5$ and (b) those with $1.3 < E < 1.5$. A computer search for Σ_2 relations among the first group was made. In addition, the

program searched for Σ_2 relations between each reflexion in the second group and those of the first, in order to facilitate the extension of phases belonging to the second groups in terms of those in the first.

The three origin-specifying reflexions and two symbolic phases which were assigned are given below.

h	k	l	$ E $	Phase	No. of Σ_2 relations for each reflexion
7	2	4	3·2	+	29
2	4	3	2·2	+	18
4	3	3	2·2	+	23
2	2	2	2·7	A	27
1	2	2	3·1	B	36

Signs of a total of 96 reflexions were determined in terms of \pm , $\pm A$, $\pm B$, $\pm AB$. The propagation of phases strongly indicated that sign of symbol $A \sim -$ and $B \sim -$.

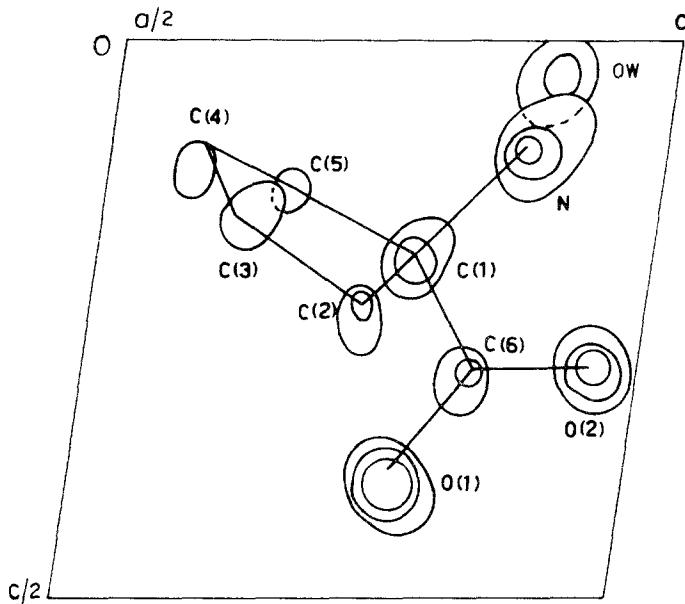


FIG. 1. E map computed with 96 signs.

An E map was calculated for the 96 reflexions with $A = -$ and $B = -$, which revealed all 10 non-hydrogen atoms, figure 1. All peaks except those at the ring carbon atoms were well above the average level for spurious peaks. A structure factor calculation for all 3-dimensional reflections gave an initial R factor of 48 %. Five cycles of full-matrix least-squares refinement with isotropic temperature factors for all atoms reduced R to 16 %. It was noticed at this stage that two of the ring carbon

atoms showed temperature factors comparatively larger than those of the rest of the atoms in the structure [10.5 \AA^2 for C(3) and 5.5 \AA^2 for C(4)]. All the bond lengths were normal except C(3)–C(4), which was only 1.43 \AA . These features indicated that the cyclopentane ring was possibly disordered, involving the atoms C(3) and C(4). A three-dimensional difference Fourier was computed omitting

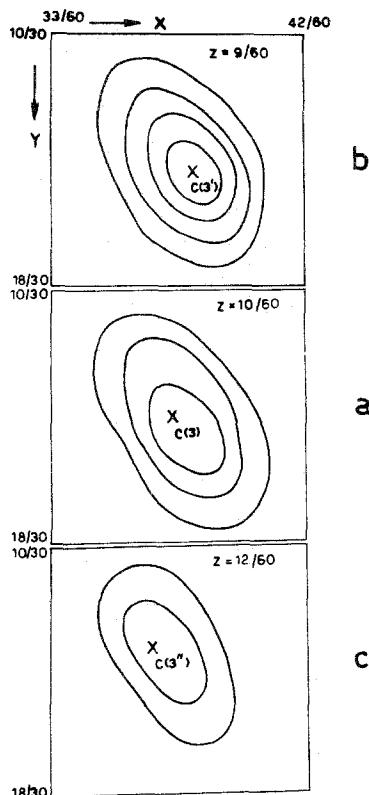


FIG. 2. Difference Fourier sections of atom C(3). Contours are at intervals of $0.5 \text{ e}/\text{\AA}^{-3}$ starting from $1.0 \text{ e}/\text{\AA}^{-3}$.

C(3) and C(4). The peak corresponding to C(4) was of height $4 \text{ e}/\text{\AA}^{-3}$ and was quite spherically symmetric while that of the peak corresponding to C(3) was rather drawn out and the peak maximum was only $2 \text{ e}/\text{\AA}^{-3}$. The difference Fourier indicated that C(4) did not show any appreciable disordered effect. Hence, another difference Fourier was computed excluding only C(3) from F_c ; sections are shown in figure 2. It was noticed that at the previously refined position of C(3) the peak height was only $2.3 \text{ e}/\text{\AA}^{-3}$ (figure 2a) while close to it a maximum of $2.6 \text{ e}/\text{\AA}^{-3}$ was observed (figure 2b), which will hereinafter be referred to as position C(3'). With

the new position C(3'), the bond length C(3')—C(4) showed marked improvement ($=1.54 \text{ \AA}$). In the extended peak around carbon atom C(3), another position C(3'') (figure 2c) could be found which again gave good bond length (C(3'')—C(4) = 1.54 \AA). Positions C(3') and C(3'') could be considered to be two alternative sites for the ring carbon atom C(3), and it is seen from figure 2 that these positions were observed in sections along Z on either side of the previously refined position of C(3) and separated by a distance of 0.74 \AA . From peak heights, position C(3') had a larger occupancy factor ('predominant' position) than C(3'') ('alternative' position). The occupancy parameter of the two sites of the atom C(3) was determined in the same way as in the case of 1-aminocycloheptane carboxylic acid hydrobromide monohydrate (Chacko *et al.* 1971a). If $(1 - x)F_1$ represents a struc-

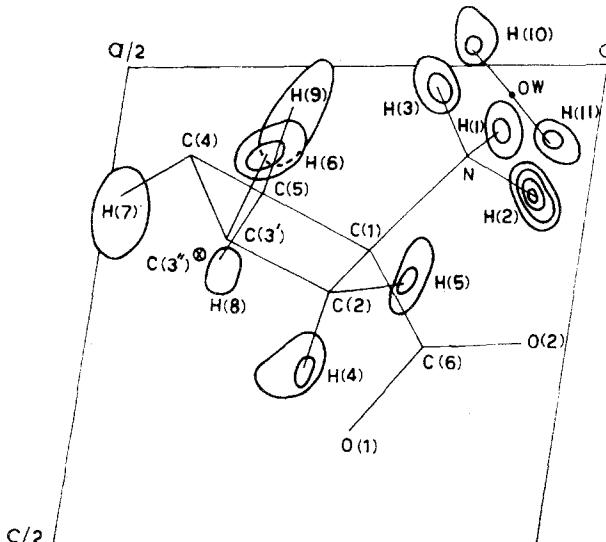


FIG. 3. Composite diagram of the difference Fourier showing the hydrogen positions. Contours are at intervals of $0.1 \text{ e}/\text{\AA}^3$ starting from $0.2 \text{ e}/\text{\AA}^3$.

ture factor for the 'predominant' position C(3'), where $(1 - x)$ represent its occupancy, and xF_2 represents the structure factor for the 'alternative' position C(3''), then the total structure factor F can be written as $(1 - x)F_1 + xF_2 + F_3 = F$ where F_3 corresponds to the rest of the atoms in the structure. The value of x was varied and gave a minimum R factor of 15.4% for $x = 0.41$. The temperature factors for the two sites were each 5.0 \AA^2 .

A difference Fourier was computed at this stage in order to locate the hydrogen atom positions. The hydrogen positions were fixed theoretically and the difference Fourier scanned near the expected positions. The hydrogens which are involved in the hydrogen bonding scheme (namely the three hydrogens from the protonated

amino nitrogen and the water molecule) appeared close to the theoretically expected positions with peak heights between 0.3 to $0.6 \text{ e}\text{\AA}^{-3}$. Some of the hydrogens of the cyclopentane ring were also seen, although the peaks corresponding to these were slightly smeared mainly owing to the disorder effect of the pentane ring. The composite diagram of the hydrogen positions is figure 3.

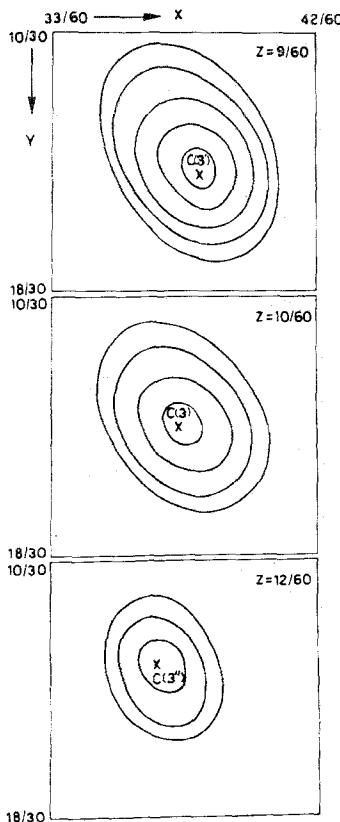


FIG. 4. Final Fourier sections of atoms C(3). Contours are at intervals of $0.5 \text{ e}\text{\AA}^{-3}$ starting from $1.0 \text{ e}\text{\AA}^{-3}$.

An attempt to refine the positions C(3') and C(3'') with occupancy factors determined as given above together with the rest of the atoms in the structure proved futile, since it resulted in bonds involving these two atoms again showing abnormalities. The reason for this may be due to the fact that the occupancy factor for the split atom position could not be refined with position and temperature factors using the program available. Also, the distance of separation of C(3') and C(3'') was just 0.74 \AA , and with the present data it is not possible to refine over such

a small distance of separation. In successive cycles, these positions were not refined. The weighting scheme applied was that of Cruickshank *et al.* (1961) in the form $w = 1/(a + F_0 + cF_0^2)$ where the constants a and c were given values equal to 8.0 and 0.04 respectively. Anisotropic thermal corrections were applied to all ordered atoms and overall scale factors were alone refined. Three cycles of full-matrix refinement reduced the R factor to 12.5%. The atomic shifts in the last cycle of refinement were less than 1/10th their standard deviations. Hydrogens obtained from the difference Fourier were included in the final structure factor which brought R to 12.1%. The structure factor calculations which varied the

Table 1a. *Coordinates of atoms and their standard deviations*

Atom	<i>x</i>	<i>y</i>	<i>z</i>	Occupancy
C(1)	0.7821(4)	0.2034(9)	0.1959(4)	1.00
C(2)	0.7423(6)	0.4269(10)	0.2366(6)	1.00
C(4)	0.5799(7)	0.2817(15)	0.0944(9)	1.00
C(5)	0.6633(5)	0.0974(11)	0.1349(5)	1.00
C(6)	0.8479(5)	0.0692(9)	0.3005(4)	1.00
N	0.8658(4)	0.2293(7)	0.1015(4)	1.00
O(1)	0.7875(4)	0.0216(8)	0.3846(3)	1.00
O(2)	0.9554(3)	0.0255(7)	0.2960(4)	1.00
Ow	0.8989(4)	0.2007(7)	0.0318(4)	1.00
C(3')*	0.6333	0.4861	0.1500	0.59
C(3'")*	0.6059	0.4345	0.1990	0.41

* Positions of these atoms were not refined.

Table 1b. *Anisotropic thermal parameters of atoms ($\times 10^5$)*

Atom	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
C(1)	464	1368	381	69	94	-75
C(2)	800	1603	844	989	535	-92
C(4)	700	3793	1580	390	-222	1313
C(5)	481	2335	658	-604	-160	573
C(6)	553	1177	396	-140	1	-70
N	492	1933	308	-16	185	61
O(1)	828	2832	440	-102	298	346
O(2)	568	2218	716	502	-33	702
Ow	1118	1590	801	257	-199	-477
C(3')*	$(B = 5.0 \text{ \AA}^2)$					
C(3'")*	$(B = 5.0 \text{ \AA}^2)$					

* C(3') and C(3'") were given isotropic temperature factor which were not refined.
Anisotropic temperature factor:

$$\exp(-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{12}hk + b_{13}hl + b_{23}kl)).$$

Table 1c. *Coordinates of hydrogen atoms located from the difference Fourier*

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> (\AA^2)	Bonded to
H(1)	0.892	0.070	0.070	2.1	N
H(2)	0.948	0.320	0.140	2.1	N
H(3)	0.820	0.313	0.020	2.1	N
H(4)	0.720	0.420	0.330	3.2	C(2)
H(5)	0.810	0.540	0.230	3.2	C(2)
H(6)	0.650	0.590	0.080	5.0	C(3')
H(7)	0.495	0.250	0.130	5.2	C(4)
H(8)	0.630	0.000	0.220	3.0	C(5)
H(9)	0.685	0.000	0.050	3.0	C(5)
H(10)	0.860	-0.315	-0.020	4.1	O _w
H(11)	0.950	-0.293	0.090	4.1	O _w

occupancy parameter were repeated at this stage but the *R* factor minimum (12.1%) occurred at the previously determined value of *x* (0.41). The final Fourier sections of the disordered atom C(3) (corresponding to the difference Fourier sections of figure 2) are given in figure 4 and show the split atom positions C(3') and C(3'') and the previously refined position C(3). The final refined coordinates of atoms are given in tables 1a and 1b together with the split atom positions C(3') and C(3'') of the ring carbon atom which were not refined. The hydrogens positions located from the difference Fourier are given in table 1c.

Discussion of the structure

Intramolecular features. The bond lengths and bond angles calculated from the final refined coordinates are given in figure 5, and are listed in table 2a together with the estimated standard deviations. Positions C(3') and C(3'') were not refined and hence, a discussion of bond length and bond angle involving these atoms is not fully justified. The mean estimated standard deviations in the C—C, C—N and C—O bonds are 0.009, 0.007 and 0.007 Å respectively. The C—C bond lengths of the pentane ring not involved with the disordered positions C(3') and C(3'') agree closely with the values expected and are within the accuracy of the structure. There are only two bond angles in the pentane ring which are not involved with the disordered positions; they are the angles C(5)—C(1)—C(2) and C(4)—C(5)—C(1) having values equal to 104.4 and 105.3° respectively, close to the expected angles for cyclopentane. The bond lengths and bond angles involving those hydrogen atoms observed from the difference Fourier are given in table 2b. The mean C—H, N—H and O—H bond lengths are 1.12, 1.12 and 1.00 Å respectively.

The molecule in this structure exists as a Zwitterion ($\text{NH}_3^+—\text{R}-\text{CO}_2^-$). The two C—O bond lengths are very nearly equal: C(6)—O(1) = 1.269 and C(6)—O(2) = 1.246 Å. The atoms of the carboxyl group are co-planar with C(1); a deviation

from this plane of 0.12 Å is found for the amino nitrogen. The equation of the least-squares plane is given by $0.226X + 0.846Y - 0.484Z = 4.05$. The deviations of the atoms from this plane (plane 1) are given in table 3. The C(1)—N distance of 1.516 Å is slightly larger than the average value of 1.487 Å for the C—N⁺ distance for accurately determined amino acids (Marsh and Donohue, 1967). It has been observed that in other structures of 1-aminocyclohexane carboxylic acids (Chacko *et al.* 1971*a,b*; Srikrishnan *et al.* 1971 and Chandrasekharan *et al.* 1968), the

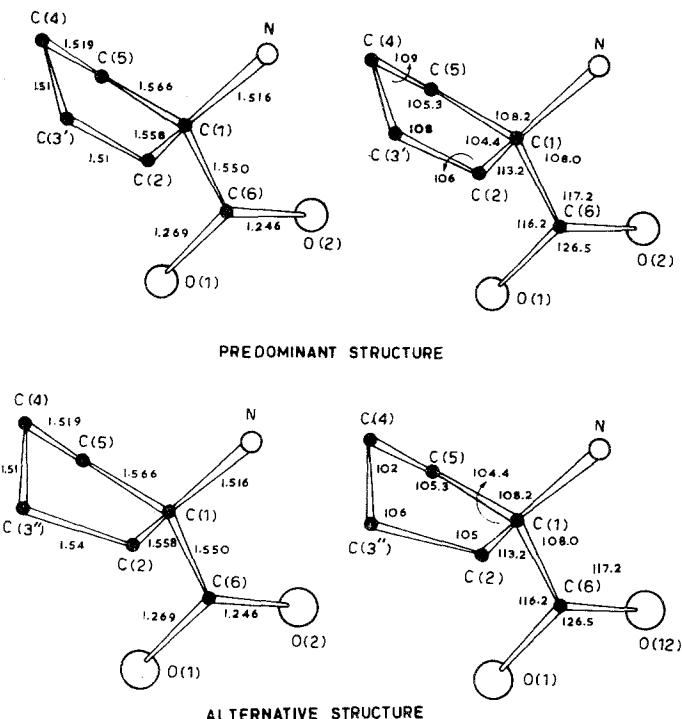


FIG. 5. Bond lengths and bond angles for the 'predominant' and 'alternative' conformations.

C—N⁺ distance is always found to be systematically larger than the above average value. Other features that have been observed in the structures of amino acids are found to exist also in this structure. The angle C(1)—C(6)—O(2) (117.2°) of the O(2) atom which is close to N is larger than the angle C(1)—C(6)—O(1) (116.2°). There are two angles ψ_1 and ψ_2 defining the disposition of the carboxyl group with respect to the amino nitrogen (Edsall *et al.* 1966). In this structure the values of ψ_1 and ψ_2 are 184° and 7° respectively.

The two alternative positions of the ring C(3) atom, namely C(3') and C(3''),

Table 2a. Bond lengths and angles and their standard deviations

Bond length (Å)	Bond angles (°)
C(1)—C(2)	1.558(8)
C(2)—C(3')	1.51 (*)
C(3')—C(4)	1.51 (*)
C(4)—C(5)	1.519(11)
C(1)—C(5)	1.566(8)
C(1)—C(6)	1.550(7)
C(1)—N	1.516(7)
C(6)—O(1)	1.269(7)
C(6)—O(2)	1.246(7)
C(2)—C(3'')	1.54 (*)
C(3'')—C(4)	1.51 (*)
	C(5)—C(1)—C(2)
	C(1)—C(2)—C(3')
	C(2)—C(3')—C(4)
	C(3')—C(4)—C(5)
	C(4)—C(5)—C(1)
	C(5)—C(1)—C(6)
	C(2)—C(1)—C(6)
	C(5)—C(1)—N
	C(6)—C(1)—N
	C(1)—C(6)—O(1)
	C(1)—C(6)—O(2)
	O(1)—C(6)—O(2)
	C(1)—C(2)—C(3'')
	C(2)—C(3'')—C(4)
	C(3'')—C(4)—C(5)
	104.4(0.6)
	106 (*)
	108 (*)
	109 (*)
	105.3(0.6)
	113.2(0.6)
	113.2(0.6)
	108.2(0.5)
	109.7(0.5)
	108.0(0.5)
	116.2(0.5)
	117.2(0.5)
	126.5(0.5)
	105 (*)
	106 (*)
	102 (*)

* Indicates that the standard deviations of these are indeterminate since the atoms C(3') and C(3'') were not refined.

Table 2b. Bond lengths and bond angles involving hydrogen atoms

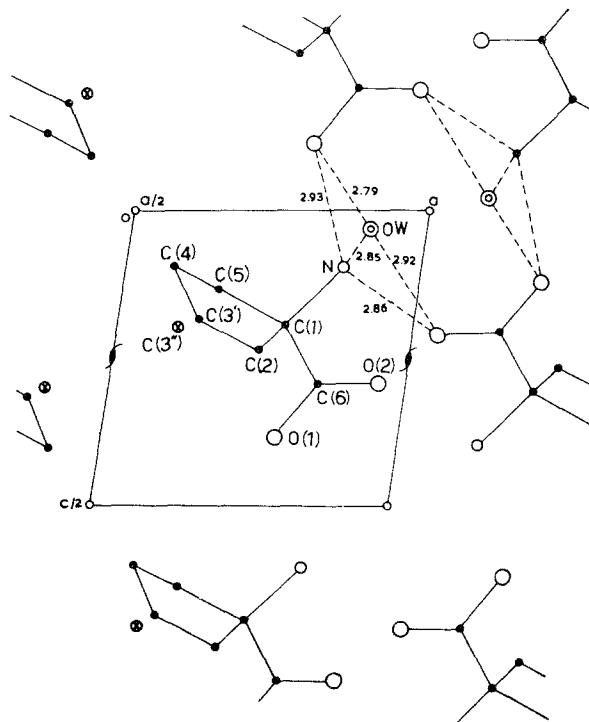
Bond length	Bond angle
N—H(1)	1.11 Å
N—H(2)	1.12
N—H(3)	1.12
C(2)—H(4)	1.11
C(2)—H(5)	1.05
C(3')—H(6)	1.06
C(4)—H(7)	1.10
C(5)—H(8)	1.23
C(5)—H(9)	1.18
Ow—H(10)	0.99
Ow—H(11)	1.00
	C(1)—N—H(1)
	C(1)—N—H(2)
	C(1)—N—H(3)
	C(1)—C(2)—H(4)
	C(1)—C(2)—H(5)
	C(3')—C(2)—H(4)
	C(3')—C(2)—H(5)
	C(2)—C(3')—H(6)
	C(4)—C(3')—H(6)
	C(3')—C(4)—H(7)
	C(5)—C(4)—H(7)
	C(4)—C(5)—H(8)
	C(4)—C(5)—H(9)
	C(1)—C(5)—H(8)
	C(1)—C(5)—H(9)
	H(10)—Ow—H(11)
	110°
	110
	111
	111
	110
	111
	109
	115
	108
	109
	107
	112
	110
	102
	108
	98

give an admixture of two conformations similar to those observed for 1-amino-cycloheptane carboxylic acid hydrobromide monohydrate (Chacko *et al.* 1971a). In the present structure, the two conformations are of the envelope type as given by Pitzer & Donath (1959) and Hendrickson (1961) for pure cyclopentane. The

Table 3. Least squares planes for the carboxyl group and the ring carbons

Plane 1 through C(1), C(6), O(1), O(2)		Plane 2 through C(2), C(3'), C(4), C(5)		Plane 3 through C(1), C(2), C(3''), C(5)	
Atom	Deviation	Atom	Deviation	Atom	Deviation
C(1)	0.003 Å	C(2)	-0.020 Å	C(1)	-0.004 Å
C(6)	-0.014	C(3')	0.036	C(2)	0.004
O(1)	0.005	C(4)	-0.040	C(3'')	-0.003
O(2)	0.005	C(5)	0.023	C(5)	0.002
N	0.12	C(1)	0.44	C(4)	0.61

pentane ring with atoms C(1), C(2), C(3'), C(4) and C(5) could be considered as a 'predominant' conformation of the envelope type with atoms C(2), C(3'), C(4) and C(5) very nearly coplanar, but with C(1) deviating from the plane by 0.44 Å. The equation to this plane is given by $0.653X + 0.153Y - 0.742Z = 3.645$; the

FIG. 6. View of the structure projected down *b*.

deviations of atoms from this plane (plane 2) are given in table 3. The 'alternative' conformation with the atoms C(1), C(2), C(3''), C(4) and C(5) is also of the envelope type with C(4) atom deviating from the mean plane formed by the other four by 0.61 Å; the equation of the plane is $0.267X + 0.389Y - 0.882Z = 0.851$, and the deviations of the atoms from this plane (plane 3) are given in table 3. It appears, however, in the case of 1-aminocyclopentane carboxylic acid hydrobromide (Chandrasekharan *et al.* 1968) that the conformation of the cyclopentane is the half-chair form.

Intermolecular features: The structure projected down the *b* axis is shown in figure 6, which is drawn with respect to the 'predominant' position C(3') of the

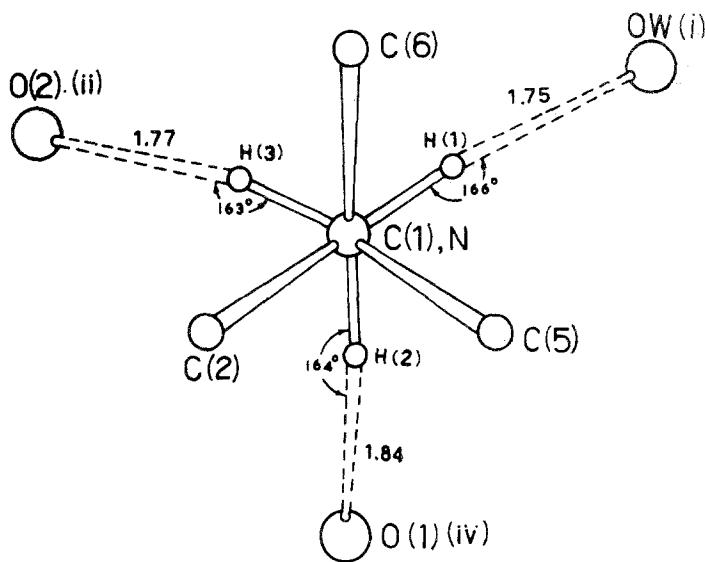


FIG. 7. Projection down the C(1)—N bond.

pentane ring (the C(3'') position is marked by \otimes). The amino nitrogen (NH_3^+) has three protons for hydrogen bonding and it forms hydrogen bonds with atoms $\text{Ow}(1)$, $\text{O}(1)(\text{iv})$ and $\text{O}(2)(\text{ii})$ at distances of 2.85, 2.93 and 2.86 Å respectively. The corresponding distances of the hydrogen positions (obtained from the difference Fourier) for the above bonds are $\text{H}(1) \cdots \text{Ow}(1) = 1.75$, $\text{H}(2) \cdots \text{O}(1)(\text{iv}) = 1.84$ and $\text{H}(3) \cdots \text{O}(2)(\text{ii}) = 1.77$ Å. These distances are much smaller than the sum of the van der Waals radii of hydrogen and oxygen, indicating that they are strong bonds. Figure 7 shows that the hydrogen bond directions are staggered with respect to the bonds covalently linked to the C(1) atom. The water oxygen atom Ow is so placed in the structure that it forms two hydrogen bonds with

Table 4. *Hydrogen bond lengths and angles*

Bond length (\AA)	Bond angle ($^{\circ}$)		
N—H(1)···O _w (i)	2.85 \AA	C(1)—N···O _w	(i)
H(1)···O _w (i)	1.75	N—H(1)···O _w	(i)
N—H(2)···O(1) (iv)	2.93	C(1)—N···O(1)	(iv)
H(2)···O(1) (iv)	1.84	N—H(2)···O(1)	(iv)
N—H(3)···O(2) (ii)	2.86	C(1)—N···O(2)	(ii)
H(3)···O(2) (ii)	1.77	N—H(3)···O(2)	(ii)
O _w —H(10)···O(1) (v)	2.79	O(1)(v)···O _w ···O(2) (iii)	98
H(10)···O(1) (v)	1.80		
O _w —H(11)···O(2) (iii)	2.92		
H(11)···O(2) (iii)	1.93		

Symmetry code:

(i)	x	y	z
(ii)	$2 - x$	$\frac{1}{2} + y$	$\frac{1}{2} - z$
(iii)	$2 - x$	$y - \frac{1}{2}$	$\frac{1}{2} - z$
(iv)	x	$\frac{1}{2} - y$	$z - \frac{1}{2}$
(v)	x	$-y - \frac{1}{2}$	$z - \frac{1}{2}$

O(1)(v) and O(2)(iii) at distances of 2.79 and 2.92 \AA respectively. The corresponding distances of the hydrogens of the water molecule to the acceptor oxygen atom are H(10)···O(1)(v) = 1.80 and H(11)···O(2)(iii) = 1.93 \AA . In addition, the water molecule acts also as an acceptor of a hydrogen bond with the amino nitrogen N(i). The relevant hydrogen bond lengths and angles are given in table 4.

The authors wish to thank Professor R. Srinivasan for valuable discussions and Professor G. N. Ramachandran for his keen interest. The full-matrix least-squares refinement program used was that of Gantzel, Sparks and Trueblood, modified for the CDC 3600 computer by Zalkin, Lundgren, Liminga and Braenden. The sigma 2 search program used was that of Dr. S. T. Rao. Thanks are due to these individuals for making available their programs. This work was supported in part by Grant NB-05306 to R. Zand from the National Institute of Neurological Diseases and Stroke, National Institute of Health.

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