between N(15)—H(151) and O(14) of the molecule related by the 2_1^y axis. Thus, each molecule is hydrogen bonded to four neighbouring molecules, two related by 2_1^z (part of the helix along **z**) and two others related by 2_1^y (part of the helix along **y**). The shortest non-bonded intra- and intermolecular contacts are listed in Table 4.

The results described above indicate that the conformation of the investigated compound is rather typical for normal penicillins. The high resistance of the ureidopenicillin to β-lactamase does not seem to be due to steric hindrance in the vicinity of the β-lactam ring but is probably caused by the modification of chemical properties of the compound and its affinity with the enzyme. The introduction of the ureido in place of the carboxy group makes the molecule a substrate for transpeptidase but no longer a substrate for β-lactamase.

The authors thank Dr M. Domaradzki, Institute of Pharmaceutical Industry, Warsaw, for supplying the sample, the Freie Universität Berlin for financing the stay of Z. Dauter in Berlin and the Wissenschaftliche Rechenzentrum Berlin for calculations.

References

ABRAHAMSSON, S. HODGKIN, D. C. & MASLEN, E. N. (1963). *Biochem. J.* **86**, 514–535.

CHANEY, M. O. & JONES, N. D. (1973). *Cryst. Struct. Commun.* **2**, 367–370.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

CSÖREGH, I. & PALM, T.-B. (1977). *Acta Cryst.* B**33**, 2169–2174.

DOMARADZKI, M., MIKOŁAJCZYK, J., KAZIMIERCZAK, J., ROGOWSKA, W. & SIKORA, A. (1977). Polish Patent Appl. P 203464.

DOMIANO, P., NARDELLI, M., BALSAMO, A., MACCHIA, B. & MACCHIA, F. (1979). *Acta Cryst.* B**35**, 1363–1372.

DOMIANO, P., NARDELLI, M., BALSAMO, A., MACCHIA, B., MACCHIA, F. & MEINARDI, G. (1978). *J. Chem. Soc. Perkin Trans.* 2, pp. 1082–1087.

HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.

JOHNSON, C. K. (1976). *ORTEP* II. Report ORNL-5138, Oak Ridge National Laboratory, Tennessee.

MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J. P. (1977). *MULTAN* 77. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

PALM, T.-B. & CSÖREGH, I. (1978). *Acta Cryst.* B**34**, 138–143.

SIMON, G. L., MORIN, R. B. & DAHL, L. F. (1972). *J. Am. Chem. Soc.* **94**, 8557–8563.

STEWART, J. M. (1976). The XRAY 76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

SWEET, R. M. (1972). *Cephalosporins and Penicillins*, pp. 280–308. New York, London: Academic Press.

SWEET, R. M. & DAHL, L. F. (1970). *J. Am. Chem. Soc.* **92**, 5489–5507.

Acta Cryst. (1981). B**37**, 2183–2185

A Disordered Ring in the Molecular Structure of Cyclopentanecarboxamide (CYCLAM)

BY H. J. WINTER, H. J. HECHT AND H. BRADACZEK

Institut für Kristallographie der Freien Universität Berlin, Takustrasse 6, *D*-1000 *Berlin* 33, *Federal Republic of Germany*

(*Received* 26 *November* 1980; *accepted* 21 *April* 1981)

Abstract

$C_6H_{11}NO$, $M_r = 113{\cdot}2$, is monoclinic, space group $C2/c$ with $a = 22{\cdot}13\,(1)$, $b = 6{\cdot}392\,(5)$, $c = 9{\cdot}474\,(5)$ Å, $\beta = 100{\cdot}12\,(1)°$, $V = 1319\,(1)$ Å^3, $D_c = 1{\cdot}137\,(1)$, $D_x = 1{\cdot}10\,(5)$ Mg m^{-3}, $Z = 8$. Final $R = 0{\cdot}089$ for 925 observed reflections. The cyclopentane ring is disordered; one of the C atoms exists in two alternative positions leading to two possible conformations, both of which are approximately of the envelope type.

0567-7408/81/122183-03$01.00

Introduction

Several authors have compared the ring conformation of proline with the stable conformations of cyclopentane. A better model for proline would be cyclo-

pentanecarboxamide (CYCLAM) which can be taken as analogous to proline for the purpose of detecting substituent effects on the five-membered aliphatic ring.

Experimental

Material

CYCLAM was prepared by mixing cyclopentanecarboxylic acid with an excess of $SOCl_2$ and refluxing for 3 h. The remaining $SOCl_2$ then was removed under reduced pressure and the chloride of the acid was distilled at 325–328 K/2 kPa.

After dissolving the product in freshly distilled dioxane, dry ammonia gas was passed through the cooled solution. The mixture was filtered from the resulting precipitate of NH_4Cl and the solvent removed under reduced pressure. Chloroform was added, the solution washed with water, dried and the $CHCl_3$ evaporated in a vacuum. CYCLAM was recrystallized as colourless platelets from methanol (m.p. 446–448 K).

Analysis: calculated: C 63·60, H 9·75, N 12·40%; found: C 63·51, H 9·8, N 12·15%.

X-ray data collection

Single crystals of CYCLAM suitable for X-ray diffraction were obtained from water. Oscillation and Weissenberg photographs, taken with Cu $K\alpha$ radiation, showed the crystals to be monoclinic.

Because of the mobility of the ring atoms the data collection was carried out at approximately 183 K. The cell dimensions were measured on a four-circle diffractometer by scanning high-index axial reflections. The intensity measurements were made with the $\theta/2\theta$ scan technique, Zr-filtered Mo $K\alpha$ radiation and the five-value method on a DEC PDP 15/40 controlled Siemens single-crystal AED diffractometer (Hecht, 1976). 1174 reflections were collected and placed on approximately absolute scale by Wilson's method. 249 reflections were considered unobserved with $I < 2\sigma(I)$. Absorption corrections were not applied.

Structure determination and refinement

The structure was solved with *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1977). Full-matrix least-squares refinement with isotropic temperature factors showed that two of the ring C atoms had larger temperature factors than the others. Further investigation of the cyclopentane ring showed the same behaviour as found in 1-amino-1-cyclopentanecarboxylic acid monohydrate (Mallikarjunan, Chacko & Zand, 1972): the difference map showed for one [C(3)] of the two C atoms with high temperature factors two maxima 1 Å apart with a height of 3·2 [C(3)] and 1·6 e Å^{-3} [C(33)]. The occupancy parameters of the two positions were refined to 0·63 [C(3)] and 0·37 [C(33)]. After refinement with anisotropic temperature factors for the heavy atoms the difference map revealed all H atoms except those at C(3) and C(33). The final R [$=\sum||F_o| - |F_c||/\sum(F_o)$] was 0·089.

Results and discussion

Supercell reflections have not been detected; therefore the discussion has been restricted to a disordered ring structure. Bond distances, angles and internal rotation angles are shown in Fig. 1 and the atomic parameters are given in Table 1.*

* Lists of structure factors, anisotropic and thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36010 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

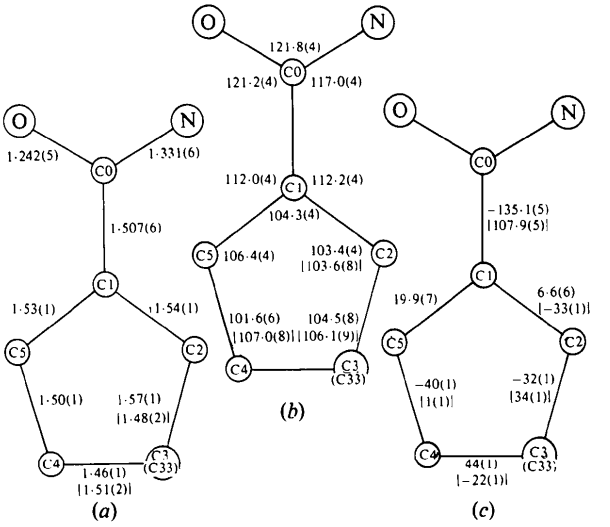

Fig. 1. (*a*) Bond lengths (Å). (*b*) Bond angles (°). (*c*) Internal rotation angles (°).

Table 1. *Atomic coordinates, population parameters and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

U_{eq} is defined according to Hamilton (1959).

	PP	x	y	z	U_{eq}/U (Å^2)
C(0)	1·0	0·3184 (2)	0·0306 (6)	0·5731 (4)	2·7 (1)
C(1)	1·0	0·3669 (2)	−0·1296 (8)	0·6277 (5)	3·7 (2)
C(2)	1·0	0·4270 (2)	−0·0922 (9)	0·5698 (7)	4·8 (2)
C(33)	0·37	0·4557 (6)	−0·302 (3)	0·577 (2)	6·2 (5)
C(3)	0·63	0·4320 (5)	−0·290 (2)	0·474 (1)	6·2 (4)
C(4)	1·0	0·4037 (3)	−0·4573 (9)	0·545 (1)	7·5 (3)
C(5)	1·0	0·3473 (3)	−0·3508 (8)	0·5778 (8)	5·6 (2)
N(1)	1·0	0·2876 (2)	0·1126 (6)	0·6682 (4)	3·1 (1)
O(1)	1·0	0·3076 (1)	0·0818 (5)	0·4446 (3)	3·1 (1)

The two ring conformations of CYCLAM, designated *C(3)* and *C(33)*, are similar to that in 1-amino-1-cyclopentanecarboxylic acid monohydrate. Both rings have approximately C_s (envelope) symmetry. The ratio of the conformations was refined to 63% *C(3)* and 37% *C(33)*. C(2) was found to deviate by 0·53 Å from the plane formed by the other four ring atoms, and is on the same side of the plane as the carboxamide C atom [C(5)–C^2-*endo*]. In the *C(3)* conformation C(4) is on the opposite side to C(0) [C(5)–C^4-*exo*] and deviates from the plane by 0·62 Å. The two conformations are shown in Fig. 2.

The carboxamide group is almost orthogonal to the plane of the cyclopentane ring. Fig. 3 shows the molecular packing.

The N atom of the amide group forms two strong hydrogen bonds, N–H(1)···O^{i} ($\frac{1}{2}-x, \frac{1}{2}-y, 1-z$) = 2·93 and N–H(2)···O^{ii} ($x, -y, \frac{1}{2}+z$) = 2·86 Å. The angles are 178·4 (i) and 177·7° (ii).

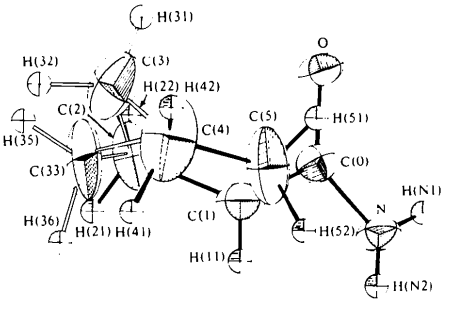

Fig. 2. The two conformations of CYCLAM.

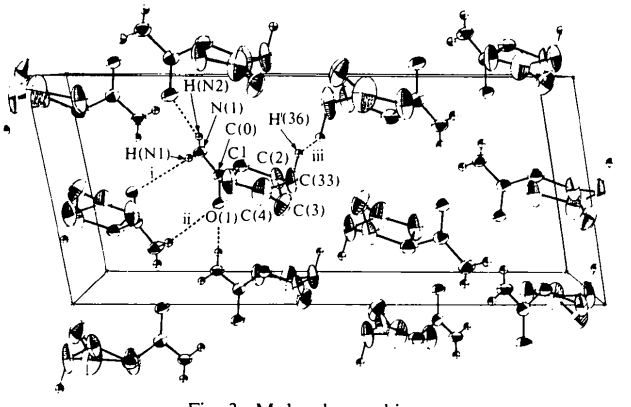

Fig. 3. Molecular packing.

All contact distances exceed the sum of the van der Waals radii, with the exception of an interaction between H(36) and H(36^{iii}) ($1-x, y, \frac{3}{2}-z$).

References

HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.

HECHT, H. J. (1976). Thesis, Freie Univ., Berlin.

MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J.-P. (1977). *MULTAN 77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain-la-Neuve, Belgium.

MALLIKARJUNAN, M., CHACKO, K. K. & ZAND, R. (1972). *J. Cryst. Mol. Struct.* **2**, 53–66.

Acta Cryst. (1981). B**37**, 2185–2189

Hydrogen Bond Studies. A Neutron Diffraction Study of the Structures of Succinic Acid at 300 and 77 K

BY JEAN-LOUIS LEVIEL* AND GEOFFROY AUVERT

Centre d'Etudes Nucléaires de Grenoble, DRF-G/Section de Résonance Magnétique, 85X, 38041 *Grenoble CEDEX, France*

AND JEAN-MICHEL SAVARIAULT

Institut Laue–Langevin, 156X, 38042 *Grenoble CEDEX, France*

(*Received* 15 *April* 1980; *accepted* 21 *April* 1981)

Abstract

A neutron diffraction study of succinic acid has been carried out at 77 and 300 K. Both unit cells are monoclinic, space group $P2_1/c$. The cell dimensions are: $a = 5{\cdot}519\ (2)$, $b = 8{\cdot}862\ (6)$, $c = 5{\cdot}101\ (2)$ Å, $\beta = 91{\cdot}59\ (4)°$, $V = 249{\cdot}39$ Å^3 at 300 K and $a = 5{\cdot}464\ (1)$, $b = 8{\cdot}766\ (3)$, $c = 5{\cdot}004\ (1)$ Å, $\beta = 93{\cdot}29\ (3)°$, $V = 239{\cdot}31$ Å^3 at 77 K. Position parameters and anisotropic temperature factors of all the atoms of half the molecule were refined by a least-squares method. The final agreement factors for the

* Université Scientifique et Médicale de Grenoble.

0567-7408/81/122185-05$01.00