Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-Aminocyclopentanecarboxamide

Qing Bao Song,[a]* You Hua Hu,[b] Tian Hua Shen[a] and Zhi Min Jin[c]

[a]College of Chemical Engineering and Materials Science, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China, [b]The Institute of Applied Chemistry, Shaoxing College of Arts and Sciences, Shaoxing 312000, Zhejiang, People's Republic of China, and [c]College of Pharmaceutical Sciences, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China

Correspondence e-mail: songqbhz@hotmail.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.106
Data-to-parameter ratio = 16.6

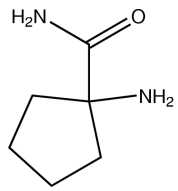
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 12 February 2004
Accepted 27 February 2004
Online 13 March 2004

Crystals of the title compound, $C_6H_{12}N_2O$, were obtained from the reduction–hydrolysis reaction of 1-amino-1-cyclopentanecarbonitrile oxalate and the structure was determined in the monoclinic space group *C*2/*c*. There are two independent molecules in the asymmetric unit and these assume different conformations. In the two cyclopentane rings, the minimum C—C bond length is 1.456 (3) Å and the maximum is 1.531 (3) Å. The weighted mean C—C bond length [1.483 (9) Å] of the two cyclopentane rings is obviously shortened in comparison with the average literature value [1.543 (18) Å] for a cyclopentane C—C bond. Hydrogen bonds of the N—H···O, N—H···N and C—H···O types are the principal intermolecular interactions.

Comment

The transformation of nitriles into carboxamides in strongly acidic media, preferably concentrated sulfuric acid (Spinale, 1996; Huszar *et al.*, 2000, 2001), raises a number of problems. To allow the reaction mixture to be stirred, sulfuric acid has to be applied in large excess. As a consequence, heating the reaction mixture to 343 K and cooling it down takes a considerable time, and keeping the reaction product for a long period in a concentrated sulfuric acid medium will cause partial decomposition, necessitating further purification steps. Since the aminocarboxamides are obtained in the form of sulfate salts, the amides have to be liberated. Neutralization of the large excess of acid means the addition of large amounts of base and also of water, in order to keep the resulting salt in solution. The resulting aminocarboxamide is well solvated and its extraction from the reaction mixture requires a minimum 40 × excess of the extracting solvent, even if the most effective, but from the aspect of health very unfavourable, chlorinated hydrocarbons are applied. These solvents can be recovered only with high losses. Against this background, we have synthesized the title compound, (I), *via* a reduction–hydrolysis reaction and present its crystal structure here.



(I)

There are two crystallographically independent molecules in the asymmetric unit of (I) and these differ in their conformation (Fig. 1). The puckering of the two cyclopentane rings is

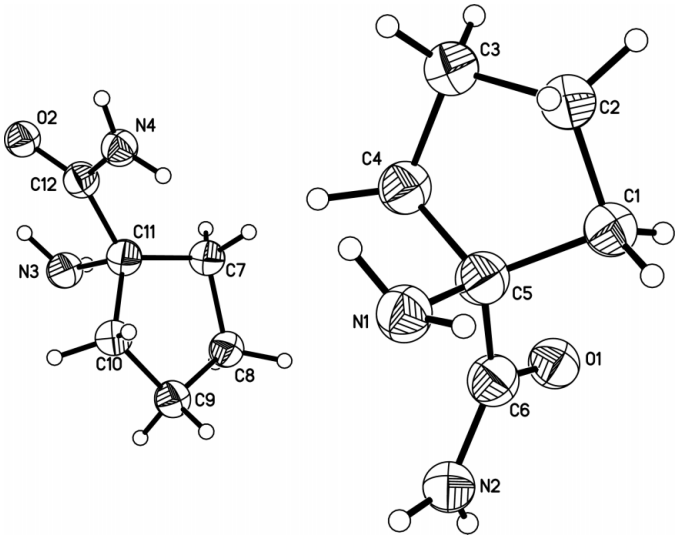


Figure 1
The asymmetric unit of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme.

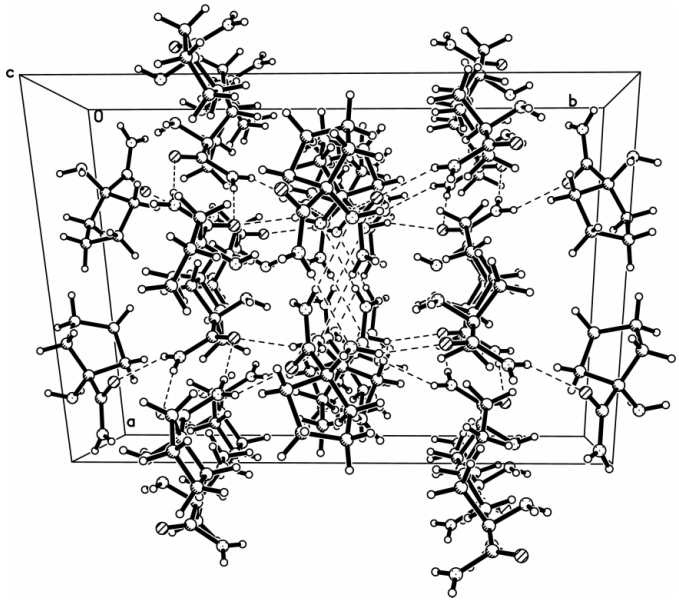


Figure 2
A packing diagram for (I), viewed down the *c* axis. Intermolecular N—H···O, N—H···N and C—H···O interactions are shown as dashed lines.

remarkably different, as indicated by the values of the Cremer & Pople (1975) total puckering amplitude parameter [Q_T = 0.408 (3) Å for the C1–C5 ring and Q_T = 0.258 (2) Å for the C7–C11 ring]. In the two cyclopentane rings, the minimum C—C bond length is 1.456 (3) Å and the maximum is 1.531 (3) Å. The weighted mean C—C bond length [1.483 (9) Å] of the two cyclopentane rings is obviously shortened in comparison with the average value [1.543 (18) Å] for a cyclopentane C—C bond (Allen *et al.*, 1987).

According to the definitions of Duax *et al.* (1976), the conformations of both rings are intermediate between twist and envelope, but for the C1–C5 ring, the local pseudo-twofold axis passes through atom C3 and the mid-point of the C1—C5 bond, and the pseudo-mirror passes through atom C1 and the mid-point of the C3—C4 bond; for the C7–C11 ring, the pseudo-twofold axis passes through atom C9 and the mid-point of the C7—C11 bond, and the pseudo-mirror passes through atom C11 and the mid-point of the C8—C9 bond.

The amino-acetamido substituents are also oriented differently in the two molecules, as shown by the following torsion angles: C4—C5—C6—N2 = −106.3 (3)°, C10—C11—C12—N4 = 74.2 (3)°, C1—C5—C6—N2 = 136.3 (3)°, C7—C11—C12—N4 = −71.5 (3)°, N1—C5—C6—N2 = 5.2 (4)°, N3—C11—C12—N4 = 172.8 (2)°, N1—C5—C6—O1 = −176.1 (2)° and N3—C11—C12—O2 = −8.9 (4)° (a larger selection of torsion angles is given in Table 1). The two molecules differ in their orientation of the amino group with respect to the amido group.

All these differences are caused by the different hydrogen-bonding environments of the two molecules. Hydrogen bonds of the N—H···O, N—H···N and C—H···O types are the principal intermolecular interactions in the crystal structure of (I) (Table 2 and Fig. 2).

Experimental

1-Aminocyclopentanecarbonitrile oxalate (10.0 g) was treated with concentrated sulfuric acid (15.0 ml) for 60 min with stirring. The evolution of a gas was observed and the temperature rose to 371 K. The mixture was cooled to about 313 K and poured into a mixture of ice and concentrated aqueous ammonia (8.5 ml). The suspension which formed was extracted five times with ethyl acetate. The combined organic phases were dried over sodium sulfate, filtered and concentrated. The expected product was obtained in the form of a white solid (7.5 g, 92%). Colourless single crystals of (I) suitable for diffraction analysis were obtained from a solution in ethyl acetate after one week.

Crystal data

$C_6H_{12}N_2O$
M_r = 128.18
Monoclinic, *C*2/*c*
a = 15.3005 (17) Å
b = 18.586 (2) Å
c = 12.2311 (14) Å
β = 125.509 (2)°
V = 2831.4 (5) Å^3
Z = 16
D_x = 1.203 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 869 reflections
θ = 3.3–18.9°
μ = 0.08 mm^{-1}
T = 293 (2) K
Prism, colourless
0.33 × 0.25 × 0.24 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.97, T_{max} = 0.98
7510 measured reflections
2772 independent reflections
1672 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 26.0°
h = −18 → 18
k = −22 → 18
l = −13 → 15

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.106
S = 0.94
2772 reflections
115 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.09 e Å^{-3}
$\Delta\rho_{min}$ = −0.10 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C5	1.461 (3)	C7—C11	1.459 (3)
C1—C2	1.464 (3)	C7—C8	1.531 (3)
C2—C3	1.515 (3)	C8—C9	1.462 (3)
C3—C4	1.486 (3)	C9—C10	1.506 (3)
C4—C5	1.493 (3)	C10—C11	1.456 (3)
C5—N1	1.470 (3)	C11—N3	1.353 (3)
C5—C6	1.538 (3)	C11—C12	1.449 (3)
C6—O1	1.220 (2)	C12—O2	1.225 (3)
C6—N2	1.300 (3)	C12—N4	1.308 (3)
C5—C1—C2	94.32 (17)	C11—C7—C8	104.64 (17)
C1—C2—C3	110.14 (19)	C9—C8—C7	106.88 (17)
C4—C3—C2	104.97 (18)	C8—C9—C10	107.02 (18)
C3—C4—C5	99.79 (18)	C11—C10—C9	106.47 (18)
C1—C5—N1	117.3 (2)	N3—C11—C12	89.66 (18)
C1—C5—C4	111.58 (18)	N3—C11—C10	97.47 (18)
N1—C5—C4	104.20 (17)	C12—C11—C10	122.19 (19)
C1—C5—C6	107.11 (18)	N3—C11—C7	111.65 (19)
N1—C5—C6	113.62 (18)	C12—C11—C7	122.69 (19)
C4—C5—C6	102.05 (19)	C10—C11—C7	107.47 (17)
O1—C6—N2	104.0 (2)	O2—C12—N4	117.2 (2)
O1—C6—C5	119.7 (2)	O2—C12—C11	134.7 (2)
N2—C6—C5	136.3 (2)	N4—C12—C11	108.1 (2)
C2—C1—C5—C4	44.5 (2)	C8—C7—C11—N3	−77.9 (2)
C5—C1—C2—C3	−36.2 (3)	C11—C7—C8—C9	−19.8 (2)
C2—C1—C5—N1	−75.5 (2)	C8—C7—C11—C10	27.8 (2)
C2—C1—C5—C6	155.45 (19)	C8—C7—C11—C12	177.8 (2)
C1—C2—C3—C4	17.9 (3)	C7—C8—C9—C10	4.6 (3)
C2—C3—C4—C5	9.3 (3)	C8—C9—C10—C11	12.4 (3)
C3—C4—C5—C1	−35.3 (3)	C9—C10—C11—C7	−25.4 (3)
C3—C4—C5—C6	−149.6 (2)	C9—C10—C11—C12	−175.6 (2)
C3—C4—C5—N1	91.9 (2)	C9—C10—C11—N3	90.1 (2)
C4—C5—C6—O1	72.4 (3)	C7—C11—C12—O2	106.8 (4)
C4—C5—C6—N2	−106.3 (3)	N3—C11—C12—O2	−8.9 (4)
C1—C5—C6—O1	−45.0 (3)	N3—C11—C12—N4	172.8 (2)
N1—C5—C6—O1	−176.1 (2)	C10—C11—C12—N4	74.2 (3)
N1—C5—C6—N2	5.2 (4)	C7—C11—C12—N4	−71.5 (3)
C1—C5—C6—N2	136.3 (3)	C10—C11—C12—O2	−107.6 (4)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*C*···O2^{i}	0.89	2.12	2.972 (2)	160
N1—H1*E*···O1^{ii}	0.89	2.51	3.312 (2)	149
N2—H2*D*···N1^{iii}	0.86	2.58	3.399 (3)	159
N3—H3*D*···O2	0.89	1.67	2.358 (2)	132
N4—H4*C*···O2^{iv}	0.86	2.13	2.983 (2)	171
N4—H4*D*···O1^{v}	0.86	2.27	3.043 (3)	149
C1—H1*A*···O1	0.97	2.38	2.755 (3)	102
C4—H4*A*···O1	0.97	2.49	2.888 (3)	105

Symmetry codes: (i) $x, y-1, z$; (ii) $x, -y, \frac{1}{2}+z$; (iii) $-x, y, \frac{3}{2}-z$; (iv) $\frac{1}{2}-x, \frac{3}{2}-y, 2-z$; (v) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$.

All H atoms were positioned geometrically and allowed to ride on their parent atoms: N—H = 0.86 and 0.89 Å, C—H = 0.97 Å, and U_{iso}(H) = 1.2U_{eq}(C,N2,N4) and 1.5U_{eq}(N1,N3).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*;.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.

Bruker (2000). *SMART*, *SAINT*, *SADABS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). *Topics in Stereochemistry*, Vol. 9, edited by E. L. Eliel & N. Allinger, pp. 271–383. New York: John Wiley.

Huszar, C., Kis-Tamas, A., Nemeth, A., Gajary, A. & Pali, L. (2000). US Patent No. 6 162 923.

Huszar, C., Kis-Tamas, A., Nemeth, A., Nad, Z., Makovi, Z., Gajary, A., Koller, E., Aranyosi, P., Gyure, K., Meszaros, I., Hari, Z. C., Supic, A., Zrinyi, I. D., Dubovszki, K., Pali, L., Karasz, A. K. & Bognar, E. (2001). US Patent No. 6 211 382.

Spinale, F. G. (1996). US Patent No. 5 541 209.