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1-(Carbamoylmethyl)cyclohexanecarboxylic acid

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.103
Data-to-parameter ratio = 15.1

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

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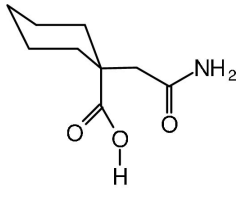
Molecules of the title compound, $C_9H_{15}NO_3$, form a two-dimensional hydrogen-bonded network, *via* O—H···O and N—H···O interactions, which runs parallel to the *bc* plane. In this structure, neither the carboxylic acid groups nor the carbamoyl groups are involved in dimer formations.

Comment

The title compound, (I), is used as an intermediate in the synthesis of biologically active heterocycles (LaRoche & Helmers, 2004). A search of the Cambridge Structural Database (Version 5.26; Allen, 2002) reveals that there are 11 structures of 1,1-disubstituted cyclohexane with a carboxylic acid group as one of the substituents. Of these, only three contain 1-cyclohexanecarboxylic acid itself. The remaining structures each contain an amino group (as the second substituent), with further attached groups on the amino N atom. There are no structures similar to 1-(carbamoylmethyl)cyclohexane.



(I)

Molecules of the title compound (Fig. 1) form a two-dimensional hydrogen-bonded network, *via* O—H···O and N—H···O interactions, which runs parallel to the *bc* plane. Hydrogen-bonding associations are listed in Table 1. The carboxylic OH group hydrogen bonds to the carbamoyl O atom of an adjacent molecule while the amino group of that molecule, in return, hydrogen bonds with the carbamoyl O atom of the first molecule. These two associations form a hydrogen-bonded ring motif [$R_2^2(11)$ graph set (Etter, 1990)] that, when repeated, propagates the hydrogen-bonding network in the *b*-axis direction. An N—H···O association between the second amino H atom and an adjacent carboxyl carbonyl O atom in the *c*-axis direction generates the two-dimensional network. Interestingly, in this structure, neither the carboxylic acid groups nor the carbamoyl groups are involved in $R_2^2(8)$ graph-set dimer formations, with like groups or with each other.

Experimental

Cyclohexanone (1.04 g, 10 mmol) was treated with ethyl cyanoacetate (1.06 g, 10 mmol) in the presence of NaOH (5 ml, 10%

aqueous solution). The resultant compound was further treated with NaCN (0.49 g, 10 mmol) in ethanol (5 ml), and hydrolysed to obtain the title compound. Crystals were grown from methanol.

Crystal data

$C_9H_{15}NO_3$
M_r = 185.22
Monoclinic, $P2_1/c$
a = 13.4973 (5) Å
b = 8.0905 (2) Å
c = 8.8358 (3) Å
β = 102.627 (2)°
V = 941.53 (5) Å^3
Z = 4
D_x = 1.307 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2268 reflections
θ = 2.9–27.5°
μ = 0.10 mm^{-1}
T = 120 (2) K
Prism, colourless
0.65 × 0.30 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.939, T_{max} = 0.990
10959 measured reflections
1842 independent reflections
1627 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 26.0°
$h = -16 \rightarrow 16$
$k = -9 \rightarrow 9$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.103
S = 1.05
1842 reflections
122 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.4339P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.048 (6)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O1^{i}	0.963 (18)	1.640 (19)	2.5829 (13)	165 (2)
N1—H1···O1^{ii}	0.88	2.21	3.0680 (15)	164
N1—H2···O2^{iii}	0.88	2.12	2.9635 (15)	162

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

The carboxyl H atom was located in a difference Fourier synthesis and its positional parameters were refined. Other H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.99 Å and N—H distances of 0.88 Å. The isotropic displacement parameters for all H atoms were set equal to $1.25U_{eq}$ of the carrier atom.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

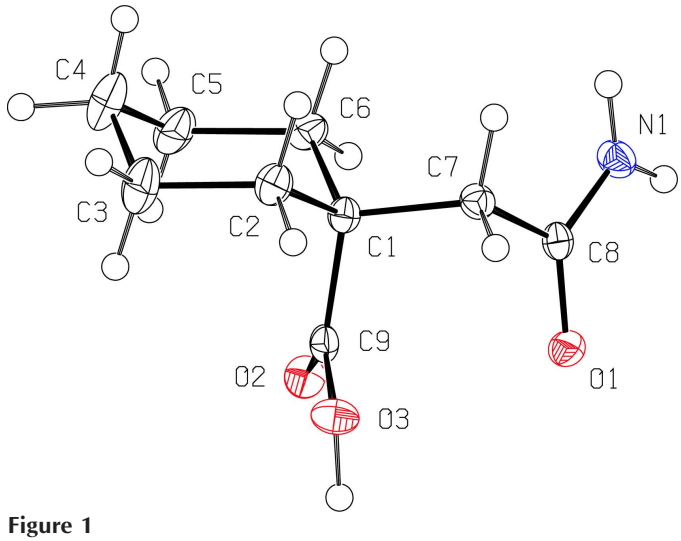


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius.

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References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Fletcher, D. A., McMeeking, R. F. & Parkin, D. J. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.
Hooft, R. W. W. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
LaRoche, S. M. & Helmers, S. L. (2004). *JAMA*, **291**, 605–614.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

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$\beta = 102.627$ (2)°
$V = 941.53$ (5) Å^3
$Z = 4$
$F(000) = 400$
$D_x = 1.307$ Mg m^{-3}
Melting point: 437 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2268 reflections
$\theta = 2.9$–$27.5°$
$\mu = 0.10$ mm^{-1}
$T = 120$ K
Prism, colourless
0.65 × 0.30 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
10 cm confocal mirrors monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.939$, $T_{max} = 0.990$
10959 measured reflections
1842 independent reflections
1627 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 26.0°$, $\theta_{min} = 3.5°$
$h = -16\rightarrow16$
$k = -9\rightarrow9$
$l = -10\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.103$
$S = 1.05$
1842 reflections
122 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.4339P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.048 (6)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.01177 (7)	0.16623 (11)	0.19598 (11)	0.0192 (3)

O2	0.18173 (7)	−0.03683 (12)	0.38790 (11)	0.0214 (3)
O3	0.11848 (8)	−0.18613 (12)	0.17583 (11)	0.0224 (3)
H3	0.0775 (13)	−0.237 (2)	0.239 (2)	0.028*
N1	0.07782 (9)	0.40190 (14)	0.12383 (13)	0.0202 (3)
H1	0.0420	0.4629	0.1748	0.025*
H2	0.1191	0.4493	0.0724	0.025*
C1	0.22107 (9)	0.04376 (16)	0.14274 (15)	0.0157 (3)
C2	0.27157 (10)	−0.06508 (17)	0.03825 (16)	0.0207 (3)
H21	0.2217	−0.1471	−0.0157	0.026*
H22	0.2921	0.0050	−0.0415	0.026*
C3	0.36471 (11)	−0.1557 (2)	0.1304 (2)	0.0310 (4)
H31	0.3966	−0.2201	0.0585	0.039*
H32	0.3434	−0.2340	0.2033	0.039*
C4	0.44223 (11)	−0.0342 (2)	0.2213 (2)	0.0354 (4)
H41	0.4999	−0.0963	0.2844	0.044*
H42	0.4688	0.0368	0.1480	0.044*
C5	0.39417 (11)	0.0735 (2)	0.32693 (18)	0.0271 (4)
H51	0.4443	0.1561	0.3789	0.034*
H52	0.3751	0.0037	0.4081	0.034*
C6	0.29991 (10)	0.16275 (17)	0.23608 (16)	0.0199 (3)
H61	0.3207	0.2429	0.1642	0.025*
H62	0.2683	0.2255	0.3094	0.025*
C7	0.13317 (10)	0.14047 (17)	0.03674 (15)	0.0169 (3)
H71	0.1622	0.2166	−0.0298	0.021*
H72	0.0889	0.0611	−0.0320	0.021*
C8	0.06951 (9)	0.23871 (17)	0.12452 (14)	0.0159 (3)
C9	0.17348 (10)	−0.06333 (16)	0.25029 (15)	0.0161 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0178 (5)	0.0183 (5)	0.0229 (5)	0.0012 (4)	0.0078 (4)	0.0003 (4)
O2	0.0227 (5)	0.0249 (5)	0.0166 (5)	0.0010 (4)	0.0041 (4)	−0.0012 (4)
O3	0.0279 (6)	0.0187 (5)	0.0218 (5)	−0.0066 (4)	0.0077 (4)	−0.0022 (4)
N1	0.0241 (6)	0.0163 (6)	0.0219 (6)	0.0026 (5)	0.0088 (5)	0.0015 (5)
C1	0.0153 (6)	0.0155 (6)	0.0161 (7)	0.0014 (5)	0.0031 (5)	−0.0009 (5)
C2	0.0200 (7)	0.0217 (7)	0.0216 (7)	0.0030 (5)	0.0069 (6)	−0.0030 (6)
C3	0.0230 (8)	0.0316 (9)	0.0376 (9)	0.0101 (6)	0.0051 (7)	−0.0063 (7)
C4	0.0167 (7)	0.0444 (10)	0.0433 (10)	0.0075 (7)	0.0024 (7)	−0.0053 (8)
C5	0.0166 (7)	0.0317 (8)	0.0302 (8)	−0.0026 (6)	−0.0009 (6)	−0.0044 (6)
C6	0.0174 (7)	0.0201 (7)	0.0224 (7)	−0.0027 (5)	0.0046 (6)	−0.0025 (5)
C7	0.0184 (7)	0.0176 (7)	0.0151 (6)	0.0011 (5)	0.0042 (5)	0.0013 (5)
C8	0.0151 (6)	0.0181 (7)	0.0126 (6)	0.0015 (5)	−0.0009 (5)	0.0010 (5)
C9	0.0145 (6)	0.0145 (6)	0.0187 (7)	0.0038 (5)	0.0022 (5)	0.0003 (5)

Geometric parameters (Å, º)

O1—C8	1.2510 (16)	C3—C4	1.529 (2)
O2—C9	1.2156 (16)	C3—H31	0.99
O3—C9	1.3257 (16)	C3—H32	0.99
O3—H3	0.963 (18)	C4—C5	1.522 (2)
N1—C8	1.3252 (18)	C4—H41	0.99
N1—H1	0.88	C4—H42	0.99
N1—H2	0.88	C5—C6	1.529 (2)
C1—C9	1.5274 (18)	C5—H51	0.99
C1—C6	1.5348 (18)	C5—H52	0.99
C1—C2	1.5387 (18)	C6—H61	0.99
C1—C7	1.5528 (17)	C6—H62	0.99
C2—C3	1.528 (2)	C7—C8	1.5043 (18)
C2—H21	0.99	C7—H71	0.99
C2—H22	0.99	C7—H72	0.99
C9—O3—H3	111.3 (10)	C3—C4—H42	109.5
C8—N1—H1	120.0	H41—C4—H42	108.1
C8—N1—H2	120.0	C4—C5—C6	111.46 (12)
H1—N1—H2	120.0	C4—C5—H51	109.3
C9—C1—C6	110.97 (11)	C6—C5—H51	109.3
C9—C1—C2	110.51 (10)	C4—C5—H52	109.3
C6—C1—C2	109.59 (10)	C6—C5—H52	109.3
C9—C1—C7	106.94 (10)	H51—C5—H52	108.0
C6—C1—C7	110.90 (10)	C5—C6—C1	112.67 (11)
C2—C1—C7	107.86 (10)	C5—C6—H61	109.1
C3—C2—C1	112.06 (11)	C1—C6—H61	109.1
C3—C2—H21	109.2	C5—C6—H62	109.1
C1—C2—H21	109.2	C1—C6—H62	109.1
C3—C2—H22	109.2	H61—C6—H62	107.8
C1—C2—H22	109.2	C8—C7—C1	113.73 (10)
H21—C2—H22	107.9	C8—C7—H71	108.8
C2—C3—C4	111.14 (13)	C1—C7—H71	108.8
C2—C3—H31	109.4	C8—C7—H72	108.8
C4—C3—H31	109.4	C1—C7—H72	108.8
C2—C3—H32	109.4	H71—C7—H72	107.7
C4—C3—H32	109.4	O1—C8—N1	122.14 (12)
H31—C3—H32	108.0	O1—C8—C7	120.10 (12)
C5—C4—C3	110.83 (12)	N1—C8—C7	117.75 (12)
C5—C4—H41	109.5	O2—C9—O3	123.19 (12)
C3—C4—H41	109.5	O2—C9—C1	124.28 (12)
C5—C4—H42	109.5	O3—C9—C1	112.48 (11)
C9—C1—C2—C3	−67.91 (14)	C6—C1—C7—C8	−66.21 (14)
C6—C1—C2—C3	54.69 (15)	C2—C1—C7—C8	173.78 (11)
C7—C1—C2—C3	175.52 (12)	C1—C7—C8—O1	−70.50 (15)
C1—C2—C3—C4	−56.56 (17)	C1—C7—C8—N1	108.96 (13)

C2—C3—C4—C5	56.00 (18)	C6—C1—C9—O2	13.30 (17)
C3—C4—C5—C6	−55.11 (18)	C2—C1—C9—O2	135.09 (13)
C4—C5—C6—C1	55.11 (16)	C7—C1—C9—O2	−107.76 (14)
C9—C1—C6—C5	68.36 (14)	C6—C1—C9—O3	−169.07 (11)
C2—C1—C6—C5	−53.96 (15)	C2—C1—C9—O3	−47.28 (14)
C7—C1—C6—C5	−172.93 (11)	C7—C1—C9—O3	69.87 (13)
C9—C1—C7—C8	54.90 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O1^{i}	0.963 (18)	1.640 (19)	2.5829 (13)	165 (2)
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