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1-Hydroxycyclobutane-1-carboxylic acid

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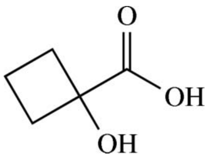
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.029; wR factor = 0.079; data-to-parameter ratio = 8.0.

The title compound, $C_5H_8O_3$, was prepared as a potentially chelating molecule bearing the conformationally rigid cyclobutane group. The cyclobutane ring is arranged perpendicular to the carboxyl group. In the polar crystal structure, each molecule exhibits four hydrogen-bonded contacts to neighbouring molecules. Instead of the formation of typical carboxylic acid dimers, an infinite hydrogen-bonded chain with alternating O–H and O═C—O—H units is observed.

Related literature

For the synthesis, see Becker *et al.* (2001). The pattern of hydrogen bonding differs from that observed for the homologues, 1-hydroxycyclopropane-1-carboxylic acid (Betz & Klüfers, 2007*a*) and 1-hydroxycyclopentane-1-carboxylic acid (Betz & Klüfers, 2007*b*). For a similar case of high displacement parameters in a cyclobutane ring, see Muranishi & Okabe (2004). For hydrogen-bonding graph-set notation, see Bernstein *et al.* (1995).



Experimental

Crystal data

$C_5H_8O_3$
M_r = 116.12
Trigonal, $R3c$
a = 10.158 (5) Å
c = 28.747 (5) Å
V = 2568.9 (18) Å^3
Z = 18
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 (2) K
0.25 × 0.20 × 0.16 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
2401 measured reflections
657 independent reflections
629 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.013

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.079
S = 1.07
657 reflections
82 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max}$ = 0.15 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.11 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O12$^{\rm i}$	0.85 (4)	2.07 (4)	2.895 (2)	163 (3)
O2—H2···O2$^{\rm i}$	0.85 (4)	2.51 (3)	3.037 (3)	121 (3)
O11—H11···O2$^{\rm ii}$	0.92 (4)	1.74 (4)	2.6488 (19)	170 (4)

Symmetry codes: (i) $-x+y, -x, z$; (ii) $-y+\frac{1}{3}, -x+\frac{2}{3}, z+\frac{1}{6}$.

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2033).

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supplementary materials

Acta Cryst. (2007). E**63**, o4032 [**doi:10.1107/S1600536807043747**]

1-Hydroxycyclobutane-1-carboxylic acid

R. Betz and **P. Klüfers**

Comment

The title compound, $C_5H_8O_3$, was prepared as a potential chelating molecule bearing the conformational rigid cyclobutane group.

The cyclobutane ring adopts a perpendicular orientation to the carboxyl group (Fig. 1). One carbon atom of the cyclobutane ring shows a comparatively high anisotropic displacement parameter – a finding, which is in agreement with the results obtained for a palladium complex including cyclobutane moieties (Muranishi & Okabe, 2004). The observed bond lengths are in agreement with valence considerations. Intermolecular hydrogen bonds determine the crystal structure. Due to the trigonal 3-axes symmetry, trimer ring systems with graph set notation $R_3^3(5)$ and infinite chains C(5) (Bernstein *et al.*, 1995) form the three-dimensional network. The formation of dimeric units upon hydrogen-bond formation – as is apparent in the structures of 1-hydroxycyclopropane-1-carboxylic acid (Betz & Klüfers, 2007*a*) and the cyclopentane analogue (Betz & Klüfers, 2007*b*) – is not observed. Instead, infinite bonding sequences of the type $(\cdots O{=}C—O–H\cdots O–H\cdots)_n$ with alternating carboxy and hydroxy functions are formed.

Experimental

The title compound was prepared according to standard procedures (Becker *et al.*, 2001) upon acidic hydrolysis of the cyanohydrin of cyclobutanone. Crystals suitable for X-ray analysis were directly obtained from the crystallized reaction product.

Refinement

All H atoms were located in a difference map and refined as riding on their parent atoms. One common isotropic displacement parameter for all H atoms was refined to $U_{iso}(H) = 0.062$ (3).

Due to the absence of significant anomalous scattering the absolute structure factor, which is −1.2 with an estimated standard deviation of 1.1 for the unmerged data set, is meaningless. Thus, Friedel opposites (327 pairs) have been merged.

Figures

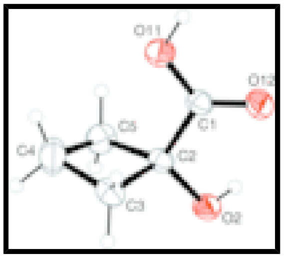

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

1-Hydroxycyclobutane-1-carboxylic acid

Crystal data

$C_5H_8O_3$

$M_r = 116.12$

Trigonal, $R3c$

Hall symbol: R 3 -2"c

$a = 10.158$ (5) Å

$b = 10.158$ (5) Å

$c = 28.747$ (5) Å

$\alpha = 90$°

$\beta = 90$°

$\gamma = 120$°

$V = 2568.9$ (18) $Å^3$

$Z = 18$

$F_{000} = 1116$

$D_x = 1.351$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 8387 reflections

$\theta = 3.1$–27.5°

$\mu = 0.11$ mm^{-1}

$T = 293$ (2) K

Block, colourless

0.25 × 0.20 × 0.16 mm

Data collection

Nonius KappaCCD diffractometer

Radiation source: rotating anode

Monochromator: MONTEL, graded multilayered X-ray optics

$T = 293(2)$ K

φ/ω–scan

Absorption correction: none

2401 measured reflections

657 independent reflections

629 reflections with $I > 2\sigma(I)$

$R_{int} = 0.013$

$\theta_{max} = 27.5$°

$\theta_{min} = 3.7$°

$h = -13 \rightarrow 13$

$k = -10 \rightarrow 10$

$l = -37 \rightarrow 37$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.079$

$S = 1.07$

657 reflections

82 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0483P)^2 + 0.8981P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.15$ e $Å^{-3}$

$\Delta\rho_{min} = -0.11$ e $Å^{-3}$

Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0040 (11)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
O2	0.18848 (17)	0.15037 (16)	0.00859 (5)	0.0401 (4)
H2	0.160 (4)	0.079 (4)	0.0286 (12)	0.070 (9)*
O11	0.2872 (2)	0.4282 (2)	0.09671 (6)	0.0553 (5)
H11	0.240 (4)	0.439 (4)	0.1225 (13)	0.075 (10)*
O12	0.06620 (18)	0.22283 (19)	0.07944 (6)	0.0501 (4)
C1	0.1989 (2)	0.3086 (2)	0.07142 (6)	0.0351 (4)
C2	0.2812 (2)	0.2928 (2)	0.02965 (6)	0.0324 (4)
C3	0.3344 (3)	0.4280 (2)	−0.00422 (7)	0.0412 (5)
H31	0.3220	0.3988	−0.0368	0.049*
H32	0.2911	0.4922	0.0023	0.049*
C4	0.4967 (3)	0.4924 (3)	0.01362 (11)	0.0609 (6)
H41	0.5287	0.5773	0.0348	0.073*
H42	0.5711	0.5169	−0.0109	0.073*
C5	0.4496 (3)	0.3412 (3)	0.03797 (9)	0.0459 (5)
H51	0.4778	0.3519	0.0706	0.055*
H52	0.4821	0.2784	0.0218	0.055*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0480 (8)	0.0331 (7)	0.0344 (7)	0.0166 (6)	0.0051 (6)	−0.0051 (6)
O11	0.0485 (9)	0.0572 (10)	0.0458 (9)	0.0155 (8)	0.0085 (7)	−0.0200 (7)
O12	0.0431 (8)	0.0510 (9)	0.0465 (8)	0.0163 (7)	0.0117 (6)	−0.0083 (7)
C1	0.0388 (10)	0.0363 (9)	0.0316 (9)	0.0199 (8)	0.0040 (7)	0.0000 (7)
C2	0.0372 (9)	0.0332 (9)	0.0291 (8)	0.0193 (7)	0.0024 (7)	−0.0013 (7)
C3	0.0492 (11)	0.0400 (10)	0.0381 (9)	0.0250 (10)	0.0093 (8)	0.0063 (8)
C4	0.0431 (13)	0.0529 (15)	0.0701 (15)	0.0115 (10)	0.0060 (12)	0.0073 (12)
C5	0.0402 (11)	0.0539 (12)	0.0503 (11)	0.0284 (10)	−0.0026 (9)	−0.0069 (9)

Geometric parameters (Å, °)

O2—C2	1.409 (2)	C3—C4	1.527 (4)
O2—H2	0.85 (4)	C3—H31	0.9700

O11—C1	1.311 (2)	C3—H32	0.9700
O11—H11	0.92 (4)	C4—C5	1.531 (4)
O12—C1	1.207 (2)	C4—H41	0.9700
C1—C2	1.517 (2)	C4—H42	0.9700
C2—C3	1.544 (3)	C5—H51	0.9700
C2—C5	1.544 (3)	C5—H52	0.9700

C2—O2—H2	110 (2)	C2—C3—H32	113.7
C1—O11—H11	114 (2)	H31—C3—H32	111.0
O12—C1—O11	124.26 (18)	C3—C4—C5	89.70 (18)
O12—C1—C2	123.42 (17)	C3—C4—H41	113.7
O11—C1—C2	112.28 (17)	C5—C4—H41	113.7
O2—C2—C1	109.16 (15)	C3—C4—H42	113.7
O2—C2—C3	113.53 (15)	C5—C4—H42	113.7
C1—C2—C3	111.77 (15)	H41—C4—H42	110.9
O2—C2—C5	117.34 (17)	C4—C5—C2	89.39 (17)
C1—C2—C5	115.15 (16)	C4—C5—H51	113.7
C3—C2—C5	88.59 (15)	C2—C5—H51	113.7
C4—C3—C2	89.54 (17)	C4—C5—H52	113.7
C4—C3—H31	113.7	C2—C5—H52	113.7
C2—C3—H31	113.7	H51—C5—H52	111.0
C4—C3—H32	113.7		

O12—C1—C2—O2	−11.5 (3)	C1—C2—C3—C4	104.08 (19)
O11—C1—C2—O2	170.40 (18)	C5—C2—C3—C4	−12.53 (19)
O12—C1—C2—C3	114.9 (2)	C2—C3—C4—C5	12.64 (18)
O11—C1—C2—C3	−63.2 (2)	C3—C4—C5—C2	−12.64 (18)
O12—C1—C2—C5	−146.0 (2)	O2—C2—C5—C4	128.41 (19)
O11—C1—C2—C5	35.9 (2)	C1—C2—C5—C4	−101.0 (2)
O2—C2—C3—C4	−131.90 (19)	C3—C2—C5—C4	12.50 (18)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O12^{i}	0.85 (4)	2.07 (4)	2.895 (2)	163 (3)
O2—H2···O2^{i}	0.85 (4)	2.51 (3)	3.037 (3)	121 (3)
O11—H11···O2^{ii}	0.92 (4)	1.74 (4)	2.6488 (19)	170 (4)

Symmetry codes: (i) −*x*+*y*, −*x*, *z*; (ii) −*y*+1/3, −*x*+2/3, *z*+1/6.

Fig. 1