organic compounds

1-Hydroxycyclopropane-1-carboxylic acid

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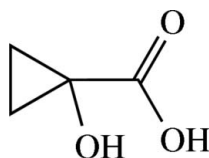Correspondence e-mail: kluef@cup.uni-muenchen.de

The title compound, $C_4H_6O_3$, was prepared as a chelating molecule bearing the conformationally rigid 1,1-cyclopropane group. The cyclopropane ring is arranged perpendicular to the carboxyl group. Intermolecular hydrogen bonds between the carboxyl groups link the molecules to form centrosymmetric dimers, which are further connected by O—H···O hydrogen bonds.

Related literature

The title compound was prepared according to standard procedures (Heine & Wendisch, 1976).

Experimental

Crystal data

$C_4H_6O_3$	V = 461.08 (3) Å^3
M_r = 102.09	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 9.0310 (3) Å	μ = 0.13 mm^{-1}
b = 4.7250 (2) Å	T = 200 (2) K
c = 12.0343 (4) Å	0.20 × 0.13 × 0.09 mm
β = 116.119 (2)°	

Data collection

Nonius KappaCCD area-detector diffractometer	1045 independent reflections
Absorption correction: none	908 reflections with $I > 2\sigma(I)$
1956 measured reflections	R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046	Only H-atom displacement parameters refined
$wR(F^2)$ = 0.127	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
S = 1.07	$\Delta\rho_{min}$ = −0.44 e Å^{-3}
1045 reflections	
65 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O2^i	0.84	2.23	2.9015 (13)	137
O11—H11···O12^{ii}	0.84	1.80	2.6336 (17)	175

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

Data collection: COLLECT (Nonius, 2004); cell refinement: SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO (Otwinowski & Minor, 1997) and SCALEPACK; 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.

References

Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Heine, H.-G. & Wendisch, D. (1976). *Liebigs Ann. Chem.* pp. 463–475.
Nonius (2004). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.

supplementary materials

1-Hydroxycyclopropane-1-carboxylic acid

R. Betz and P. Klüfers

Comment

The title compound, $C_4H_6O_3$, was prepared as a chelating molecule bearing the conformational rigid 1,1-cyclopropylene group. It was obtained upon the reaction of bromine and water on 1,2-bis(trimethylsilyloxy)-cyclobut-1,2-ene.

The cyclopropylene ring adopts a perpendicular orientation to the plane containing the atoms of the carboxyl group. All bond lengths between the central C atom and the other C atoms are found to be shorter than the typical value of 1.54 Å. Intermolecular hydrogen bonds are present in the crystal structure resulting in the formation of dimeric units.

The molecular structure (Fig. 1) shows a carboxy- and a hydroxy-group attached to the cyclopropane ring.

The molecular packing (Fig. 2) shows intermolecular hydrogen bonds between the O-bonded H atom of the carboxyl group and the double-bonded O atom of the neighbouring molecule.

Experimental

The title compound was prepared according to standard procedures (Heine & Wendisch, 1976) by the reaction of bromine and water on 1,2-bis(trimethylsilyloxy)-cyclobut-1,2-ene. Recrystallization of the product was performed from chloroform at room temperature upon free evaporation of the solvent.

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 $U_{iso}(H) = 0.052 (3) \text{ Å}^2$.
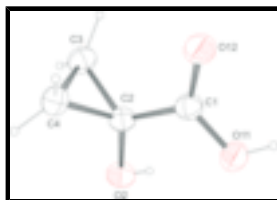
Figures

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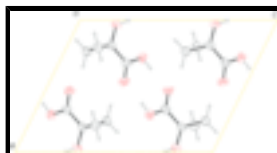

Fig. 2. The packing of (I), viewed along [010].

supplementary materials

1-Hydroxycyclopropane-1-carboxylic acid

Crystal data

$C_4H_6O_3$	$F_{000} = 216$
$M_r = 102.09$	$D_x = 1.471$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 5717 reflections
$a = 9.0310$ (3) Å	$\theta = 3.1–27.5°$
$b = 4.7250$ (2) Å	$\mu = 0.13$ mm^{-1}
$c = 12.0343$ (4) Å	$T = 200$ (2) K
$\beta = 116.119$ (2)°	Block, colourless
$V = 461.08$ (3) Å^3	$0.20 \times 0.13 \times 0.09$ mm
$Z = 4$	

Data collection

Nonius KappaCCD area-detector diffractometer	908 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	$R_{int} = 0.019$
Monochromator: MONTEL, graded multilayered X-ray optics	$\theta_{max} = 27.5°$
$T = 200(2)$ K	$\theta_{min} = 3.5°$
φ and ω scans	$h = -11 \rightarrow 11$
Absorption correction: none	$k = -6 \rightarrow 6$
1956 measured reflections	$l = -15 \rightarrow 15$
1045 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	Only H-atom displacement parameters refined
$wR(F^2) = 0.127$	$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2 + 0.294P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.07$	$(\Delta/\sigma)_{max} < 0.001$
1045 reflections	$\Delta\rho_{max} = 0.39$ e Å^{-3}
65 parameters	$\Delta\rho_{min} = -0.43$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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	0.03457 (13)	0.9329 (3)	0.19543 (11)	0.0300 (3)
H2	0.0058	0.8649	0.2474	0.052 (3)*
O11	0.28515 (15)	1.0745 (3)	0.40905 (11)	0.0372 (4)
H11	0.3673	1.1028	0.4773	0.052 (3)*
O12	0.46529 (15)	0.8034 (3)	0.37692 (12)	0.0415 (4)
C1	0.32519 (19)	0.9036 (3)	0.34354 (14)	0.0270 (4)
C2	0.19178 (18)	0.8327 (3)	0.21967 (14)	0.0252 (4)
C3	0.2062 (2)	0.5619 (4)	0.15971 (15)	0.0318 (4)
H31	0.3021	0.4385	0.2069	0.052 (3)*
H32	0.1030	0.4601	0.1071	0.052 (3)*
C4	0.2377 (2)	0.8372 (4)	0.11322 (15)	0.0305 (4)
H41	0.1540	0.9057	0.0321	0.052 (3)*
H42	0.3531	0.8841	0.1319	0.052 (3)*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0224 (6)	0.0321 (7)	0.0356 (6)	0.0007 (4)	0.0128 (5)	0.0009 (5)
O11	0.0331 (7)	0.0449 (8)	0.0304 (6)	0.0060 (5)	0.0110 (5)	−0.0078 (5)
O12	0.0273 (6)	0.0526 (9)	0.0369 (7)	0.0089 (6)	0.0069 (5)	−0.0097 (6)
C1	0.0267 (7)	0.0271 (8)	0.0293 (8)	0.0010 (6)	0.0142 (6)	0.0004 (6)
C2	0.0233 (7)	0.0238 (8)	0.0293 (8)	−0.0002 (6)	0.0124 (6)	−0.0002 (6)
C3	0.0341 (9)	0.0260 (8)	0.0350 (9)	−0.0027 (7)	0.0149 (7)	−0.0047 (7)
C4	0.0320 (8)	0.0322 (9)	0.0292 (8)	−0.0018 (7)	0.0152 (7)	−0.0009 (7)

Geometric parameters (Å, °)

O2—C2	1.4001 (18)	C2—C4	1.510 (2)
O2—H2	0.8400	C3—C4	1.492 (2)
O11—C1	1.287 (2)	C3—H31	0.9900
O11—H11	0.8400	C3—H32	0.9900
O12—C1	1.240 (2)	C4—H41	0.9900
C1—C2	1.485 (2)	C4—H42	0.9900

C2—C3	1.502 (2)		
C2—O2—H2	109.5	C4—C3—H31	117.7
C1—O11—H11	109.5	C2—C3—H31	117.7
O12—C1—O11	124.25 (15)	C4—C3—H32	117.7
O12—C1—C2	120.02 (14)	C2—C3—H32	117.7
O11—C1—C2	115.72 (14)	H31—C3—H32	114.8
O2—C2—C1	115.67 (13)	C3—C4—C2	60.04 (10)
O2—C2—C3	118.81 (13)	C3—C4—H41	117.8
C1—C2—C3	118.08 (14)	C2—C4—H41	117.8
O2—C2—C4	116.77 (13)	C3—C4—H42	117.8
C1—C2—C4	116.35 (13)	C2—C4—H42	117.8
C3—C2—C4	59.37 (11)	H41—C4—H42	114.9
C4—C3—C2	60.58 (11)		
O12—C1—C2—O2	173.88 (15)	O11—C1—C2—C4	135.37 (15)
O11—C1—C2—O2	−7.4 (2)	O2—C2—C3—C4	105.77 (16)
O12—C1—C2—C3	24.3 (2)	C1—C2—C3—C4	−105.60 (16)
O11—C1—C2—C3	−156.97 (14)	O2—C2—C4—C3	−109.19 (16)
O12—C1—C2—C4	−43.4 (2)	C1—C2—C4—C3	108.49 (16)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O2[i]	0.84	2.23	2.9015 (13)	137
O11—H11···O12[ii]	0.84	1.80	2.6336 (17)	175

Symmetry codes: (i) $-x$, $y-1/2$, $-z+1/2$; (ii) $-x+1$, $-y+2$, $-z+1$.

Fig. 1

Fig. 2