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2,2-Dicyclopropylglycolic acid

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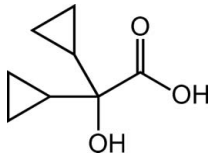
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.104; data-to-parameter ratio = 15.5.

The title compound, $C_8H_{12}O_3$, is the parent acid of a tentative ligand whose binding capability toward a metal ion is modified compared with the smaller and more hydrophilic glycolic acid. In the crystal structure, chains of homodromic rings made up of O—H–π and C═O–π co-operative sequences are lined up along [100]. The homodromic rings are at the centres of tetramers of the title compound. They are 12-membered and contain two carboxy OH, two alcohol OH and two C═O functions. The rings are connected along the chain direction by the central C—C bond of the acid. The hydrophilic chains are embedded in a lipophilic matrix of hydrocarbon residues.

Related literature

For the synthesis of the title compound, see: Zaug *et al.* (1960). For the crystal structures of some compounds bearing two cyclopropane groups bonded to an O-bonded C atom, see: Yus *et al.* (2005); Febles *et al.* (2004). For the crystal structures of 1-hydroxycarboxylic acids with the methylene unit of glycolic acid being part of smaller-sized hydrocarbon rings, see: Betz & Klüfers (2007*a*,*b*,*c*), where similar as well as different hydrogen-bonding patterns are observed. For the crystal structures of 1-hydroxycarboxylic acids in which the methylene unit of glycolic acid is substituted with sterically more demanding groups, see: Betz & Klüfers (2007*d*); Betz *et al.* (2007).



Experimental

Crystal data

$C_8H_{12}O_3$
M_r = 156.18
Triclinic, $P\overline{1}$
a = 5.8595 (3) Å
b = 7.2955 (3) Å
c = 10.5515 (5) Å
α = 92.107 (3)°
β = 100.758 (3)°
γ = 112.263 (3)°
V = 407.22 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 200 (2) K
0.16 × 0.13 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
3030 measured reflections
1594 independent reflections
1221 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.104
S = 1.04
1594 reflections
103 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H82···O3^{i}	0.84	1.83	2.6387 (16)	161
O3—H83···O1	0.84	2.15	2.6572 (16)	119
O3—H83···O1^{ii}	0.84	2.07	2.8067 (15)	146
C8—H8*B*···O2^{iii}	0.99	2.61	3.470 (2)	145

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

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: *SHELXS97* (Sheldrick, 1997); 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: BT2631).

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supplementary materials

Acta Cryst. (2007). E**63**, o4921 [**doi:10.1107/S1600536807061168**]

2,2-Dicyclopropylglycolic acid

R. Betz and P. Klüfers

Comment

2,2-Di(cyclopropyl) glycolic acid (I) was prepared as the parent acid of a potentially chelating ligand bearing two conformationally rigid cyclopropane moieties. The cyclopropane rings are oriented askew to each other (Fig. 1). Bond lengths and angles are normal (Yus *et al.*, 2005, Febles *et al.*, 2004).

Fig. 2 shows hydrophilic chains along the viewing direction embedded in a lipophilic matrix. All the contacts between the atoms of the lipophilic matrix arise at distances larger than the sum of the van-der-Waals radii.

Fig. 3 shows an individual hydrogen-bonded chain. Instead of the frequently found carboxylic-acid dimers, pairs of alcoholic OH and C=O functions are the anchoring points for a dimer. If the C1–C2 σ bond is regarded as being incapable of relaying cooperativity, and if the weakly bifurcated part of the bond from the O3–H donor is neglected, there is—contrary to a pure carboxylic acid dimer—no cooperativity in the dimer. Instead, an extended cooperative motif is formed by the mutual lateral contact of two dimers each: 12-membered, homodromic rings that contain O–H vectors as well as π-cooperativity.

Weak interactions below the sum-of-van-der-Waals-radii limit are sparse. The only contact of this kind is a non-classic C–H···O hydrogen bond that supports the hydrophilic chain along the O2···O3 contact.

Experimental

The title compound was prepared according to standard procedures (Zaug *et al.*, 1960) upon aqueous oxidation of 1,1-dicyclopropyl-prop-2-yne-1-ol. Crystals suitable for X-ray analysis were obtained upon the free evaporation of a solution of the compound in diethylether at room temperature.

Refinement

The H atoms were refined as riding on their parent atoms (U_{iso} as the 1.2-fold of the parent atom's U_{eq}). The O-bonded H atoms were refined using *SHELXL*'s electron-density-related AFIX 147 statement.

Figures

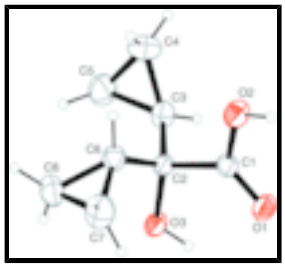

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

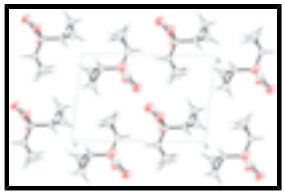

Fig. 2. The packing of (I), viewed along [−1 0 0]. One of the hydrogen-bonded chains is centered at *x*, 0, 0.

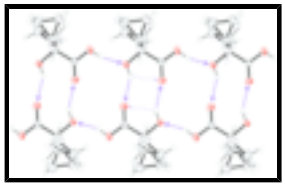

Fig. 3. An individual hydrogen-bonded chain. The [1 0 0] direction is the horizontal axis. Three dimers are shown which give rise to 12-membered homodromic rings by their mutual lateral contact. For the ring in the middle, dashed arrows point to an interaction that may be interpreted as weakly bifurcated.

2,2-Dicyclopropylglycolic acid

Crystal data

$C_8H_{12}O_3$	$Z = 2$
$M_r = 156.18$	$F_{000} = 168$
Triclinic, $P\overline{1}$	$D_x = 1.274$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.8595$ (3) Å	Cell parameters from 3975 reflections
$b = 7.2955$ (3) Å	$\theta = 3.1$–$26.0°$
$c = 10.5515$ (5) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 92.107$ (3)°	$T = 200$ (2) K
$\beta = 100.758$ (3)°	Block, colourless
$\gamma = 112.263$ (3)°	0.16 × 0.13 × 0.08 mm
$V = 407.22$ (3) Å^3	

Data collection

Nonius KappaCCD diffractometer	1221 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	$R_{int} = 0.026$
Monochromator: MONTEL, graded multilayered X-ray optics	$\theta_{max} = 26.0°$
$T = 200$(2) K	$\theta_{min} = 3.4°$
CCD; rotation images; thick slices scans	$h = -7 \rightarrow 7$
Absorption correction: none	$k = -8 \rightarrow 8$
3030 measured reflections	$l = -12 \rightarrow 12$
1594 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.039$	$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2 + 0.1023P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.104$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.19$ e Å^{-3}
1594 reflections	$\Delta\rho_{min} = -0.18$ e Å^{-3}
103 parameters	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.096 (12)
Secondary atom site location: difference Fourier map	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.45110 (17)	0.71307 (15)	0.64164 (10)	0.0310 (3)
H83	0.4429	0.6235	0.5865	0.037*
O1	0.78873 (19)	0.61259 (16)	0.55317 (11)	0.0356 (3)
O2	1.11508 (19)	0.85312 (17)	0.68539 (12)	0.0388 (4)
H82	1.1983	0.7962	0.6561	0.047*
C1	0.8731 (3)	0.7539 (2)	0.63559 (14)	0.0237 (4)
C2	0.7095 (2)	0.8385 (2)	0.69426 (14)	0.0231 (4)
C3	0.7557 (3)	1.0442 (2)	0.65394 (15)	0.0304 (4)
H3	0.7210	1.0464	0.5577	0.037*
C4	0.9573 (3)	1.2317 (2)	0.7267 (2)	0.0468 (5)
H4A	1.0446	1.3374	0.6757	0.056*
H4B	1.0650	1.2212	0.8081	0.056*
C5	0.6863 (3)	1.1889 (2)	0.72612 (19)	0.0451 (5)
H5A	0.6063	1.2680	0.6746	0.054*
H5B	0.6267	1.1517	0.8070	0.054*
C6	0.7678 (3)	0.8316 (2)	0.83998 (15)	0.0282 (4)
H6	0.9396	0.9278	0.8863	0.034*
C7	0.6769 (4)	0.6371 (3)	0.89413 (19)	0.0483 (5)
H7A	0.5677	0.5164	0.8328	0.058*
H7B	0.7929	0.6153	0.9674	0.058*
C8	0.5697 (3)	0.7866 (3)	0.91947 (17)	0.0397 (5)
H8A	0.6198	0.8577	1.0083	0.048*
H8B	0.3944	0.7589	0.8736	0.048*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0178 (5)	0.0361 (6)	0.0352 (7)	0.0091 (5)	0.0027 (5)	−0.0136 (5)
O1	0.0252 (6)	0.0375 (7)	0.0403 (7)	0.0106 (5)	0.0055 (5)	−0.0140 (5)
O2	0.0188 (6)	0.0469 (7)	0.0477 (8)	0.0137 (5)	0.0024 (5)	−0.0166 (6)
C1	0.0197 (7)	0.0277 (8)	0.0242 (8)	0.0100 (6)	0.0044 (6)	0.0013 (6)
C2	0.0167 (7)	0.0257 (8)	0.0250 (8)	0.0077 (6)	0.0026 (6)	−0.0029 (6)
C3	0.0340 (9)	0.0326 (9)	0.0284 (9)	0.0162 (7)	0.0088 (7)	0.0035 (7)
C4	0.0463 (11)	0.0298 (10)	0.0559 (12)	0.0049 (8)	0.0130 (9)	0.0055 (8)
C5	0.0573 (12)	0.0325 (10)	0.0571 (12)	0.0238 (9)	0.0257 (10)	0.0076 (8)

C6	0.0265 (8)	0.0331 (8)	0.0247 (8)	0.0121 (7)	0.0043 (6)	0.0005 (6)
C7	0.0696 (13)	0.0442 (11)	0.0415 (11)	0.0291 (10)	0.0203 (10)	0.0149 (8)
C8	0.0403 (10)	0.0526 (11)	0.0305 (9)	0.0199 (8)	0.0142 (8)	0.0051 (8)

Geometric parameters (Å, °)

O3—C2	1.4273 (16)	C4—H4A	0.9900
O3—H83	0.8400	C4—H4B	0.9900
O1—C1	1.2046 (17)	C5—H5A	0.9900
O2—C1	1.3139 (17)	C5—H5B	0.9900
O2—H82	0.8400	C6—C7	1.492 (2)
C1—C2	1.525 (2)	C6—C8	1.501 (2)
C2—C3	1.513 (2)	C6—H6	1.0000
C2—C6	1.518 (2)	C7—C8	1.489 (2)
C3—C4	1.489 (2)	C7—H7A	0.9900
C3—C5	1.497 (2)	C7—H7B	0.9900
C3—H3	1.0000	C8—H8A	0.9900
C4—C5	1.497 (3)	C8—H8B	0.9900
C2—O3—H83	109.5	C4—C5—H5A	117.8
C1—O2—H82	109.5	C3—C5—H5A	117.8
O1—C1—O2	124.35 (13)	C4—C5—H5B	117.8
O1—C1—C2	123.54 (13)	C3—C5—H5B	117.8
O2—C1—C2	112.11 (12)	H5A—C5—H5B	114.9
O3—C2—C3	108.01 (12)	C7—C6—C8	59.66 (11)
O3—C2—C6	109.26 (12)	C7—C6—C2	120.19 (14)
C3—C2—C6	114.76 (12)	C8—C6—C2	122.65 (13)
O3—C2—C1	108.04 (11)	C7—C6—H6	114.5
C3—C2—C1	109.06 (12)	C8—C6—H6	114.5
C6—C2—C1	107.53 (12)	C2—C6—H6	114.5
C4—C3—C5	60.16 (12)	C8—C7—C6	60.49 (11)
C4—C3—C2	124.26 (14)	C8—C7—H7A	117.7
C5—C3—C2	121.31 (13)	C6—C7—H7A	117.7
C4—C3—H3	113.6	C8—C7—H7B	117.7
C5—C3—H3	113.6	C6—C7—H7B	117.7
C2—C3—H3	113.6	H7A—C7—H7B	114.8
C3—C4—C5	60.20 (11)	C7—C8—C6	59.85 (11)
C3—C4—H4A	117.8	C7—C8—H8A	117.8
C5—C4—H4A	117.8	C6—C8—H8A	117.8
C3—C4—H4B	117.8	C7—C8—H8B	117.8
C5—C4—H4B	117.8	C6—C8—H8B	117.8
H4A—C4—H4B	114.9	H8A—C8—H8B	114.9
C4—C5—C3	59.63 (11)		
O1—C1—C2—O3	4.3 (2)	C1—C2—C3—C5	−161.26 (15)
O2—C1—C2—O3	−175.78 (12)	C2—C3—C4—C5	−109.52 (17)
O1—C1—C2—C3	−112.91 (16)	C2—C3—C5—C4	114.25 (17)
O2—C1—C2—C3	67.05 (16)	O3—C2—C6—C7	44.52 (18)
O1—C1—C2—C6	122.07 (16)	C3—C2—C6—C7	165.97 (14)
O2—C1—C2—C6	−57.96 (16)	C1—C2—C6—C7	−72.51 (17)
O3—C2—C3—C4	154.67 (14)	O3—C2—C6—C8	−26.75 (19)

C6—C2—C3—C4	32.5 (2)	C3—C2—C6—C8	94.70 (17)
C1—C2—C3—C4	−88.14 (18)	C1—C2—C6—C8	−143.77 (15)
O3—C2—C3—C5	81.55 (17)	C2—C6—C7—C8	−112.50 (16)
C6—C2—C3—C5	−40.6 (2)	C2—C6—C8—C7	108.48 (17)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H82···O3^{i}	0.84	1.83	2.6387 (16)	161
O3—H83···O1	0.84	2.15	2.6572 (16)	119
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C8—H8B···O2^{iii}	0.99	2.61	3.470 (2)	145

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) *x*−1, *y*, *z*.

Fig. 1

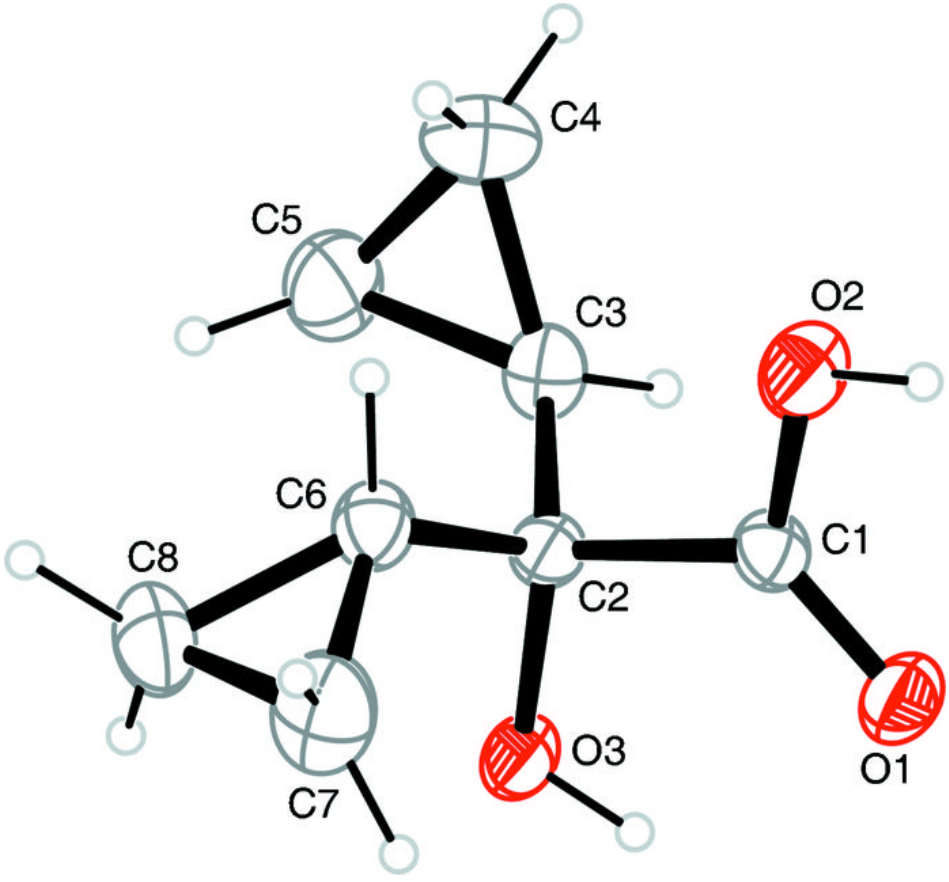

Fig. 2

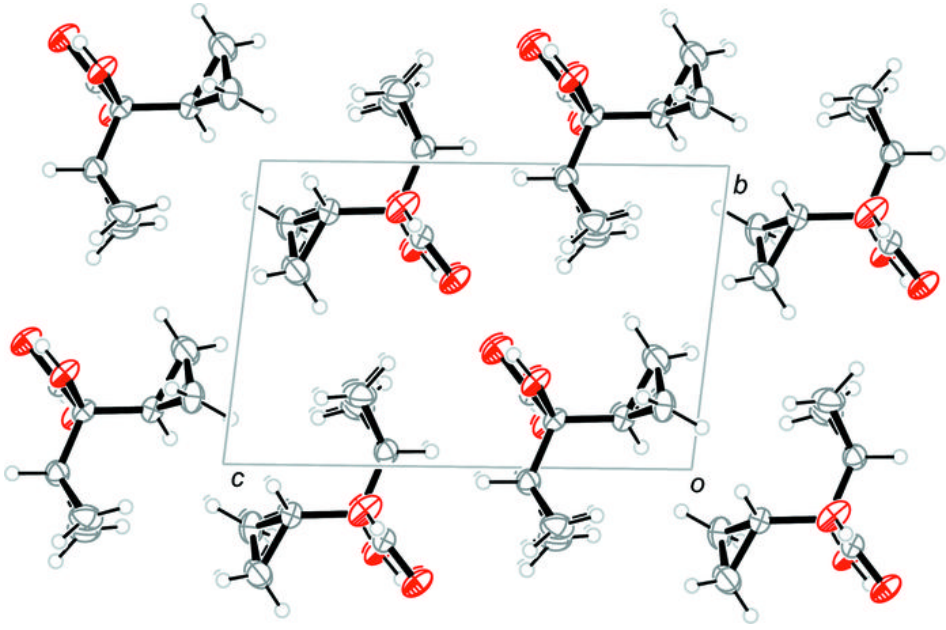

Fig. 3