Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Hydroxybicyclo[2.2.1]heptane-2-*endo*-carboxylic acid

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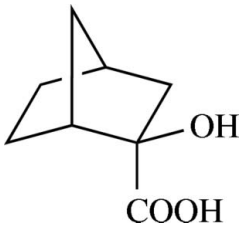
Received 24 September 2007; accepted 25 September 2007

Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.119; data-to-parameter ratio = 17.1.

In the title compound, $C_8H_{12}O_3$, the methylene function of glycolic acid is incorporated in the sterically demanding lipophilic norbornane backbone. Pairs of longer intermolecular hydroxyl OH···carboxyl O hydrogen bonds lead to the formation of centrosymmetric dimers. The dimers are connected to form sheets parallel to the *bc* plane by shorter carboxyl OH···hydroxyl O bonds. Hydrophobic contacts connect the hydrogen-bonded sheets.

Related literature

For synthesis of the title compound, see Kwart & Null (1960). For the crystal structures of 1-hydroxy-1-carboxylic acids with hydrophobic residues of similar size, see Betz & Klüfers (2007*a*,*b*,*c*). The same hydrogen-bond donor–acceptor pattern has been found for *tert*-butylglycolic acid but forming a three-dimensionally connected network instead of the two-dimensional array in the title compound (Betz *et al.*, 2007).



Experimental

Crystal data

$C_8H_{12}O_3$
M_r = 156.18
Monoclinic, $P2_1/c$
a = 11.4780 (6) Å
b = 6.8145 (3) Å
c = 10.1444 (5) Å
β = 102.506 (2)°
V = 774.64 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 200 (2) K
0.20 × 0.18 × 0.01 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
6408 measured reflections
1765 independent reflections
1314 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.119
S = 1.04
1765 reflections
103 parameters
Only H-atom displacement parameters refined
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O81^{i}	0.84	1.91	2.7447 (16)	172
O82—H82···O2^{ii}	0.84	1.83	2.6653 (15)	173

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

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*.

The authors thank Moritz Reichvilser for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2148).

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 doi:10.1107/S1600536807047071

supplementary materials

Acta Cryst. (2007). E**63**, o4185 [**doi:10.1107/S1600536807047071**]

2-Hydroxybicyclo[2.2.1]heptane-2-*endo*-carboxylic acid

R. Betz and P. Klüfers

Comment

2-Hydroxybicyclo[2.2.1]heptane-2-*endo*-carboxylic acid (2-hydroxynorbornane-2-*endo*-carboxylic acid) was prepared as the parent acid of a potentially chelating ligand bearing the sterically demanding norbornane group as a substituent. In the molecule a carboxy group and a hydroxy group are attached to the 2-position of the bicyclic norbornane framework. The carboxy groups is oriented away from the bridge-head methylene group. Bond lengths are in good agreement with literature values.

In the crystal structure, hydrophobic and hydrophilic building blocks are separated (Figure 2). In the hydrophilic blocks, a two-dimensional hydrogen-bond system is constructed by two types of bonds: pairs of longer hydroxyl-OH···carboxyl-O hydrogen bonds are found in centrosymmetric dimers. Shorter carboxyl-OH···hydroxyl-O bonds connect the dimers to form an infinite sheet (Figure 3). A three-dimensional analogue of the same connectivity pattern has been found recently for the related *tert*-butylglycolic acid (Betz *et al.*, 2007).

Experimental

The title compound was prepared according to a published procedure (Kwart & Null, 1960) by aqueous alkaline oxidation of norbornane-2-carboxylic acid with potassium permanganate. Crystals suitable for X-ray analysis were obtained by recrystallization of the crude reaction product from boiling benzene.

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) = 0.0485 (16) Å^2.

Figures

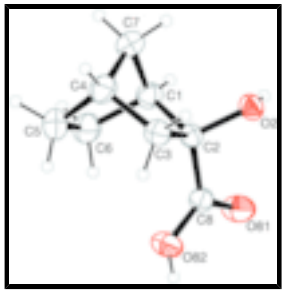

Fig. 1. The molecular structure of (I), with atom labels and anisotropic displacement ellipsoids (drawn at the 50% probability level) for non-H atoms.

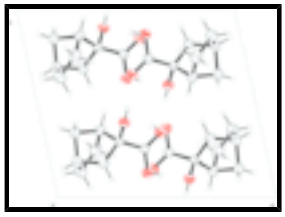

Fig. 2. Hydrophobic and hydrophilic sheets alternating along [100], viewed along [010].

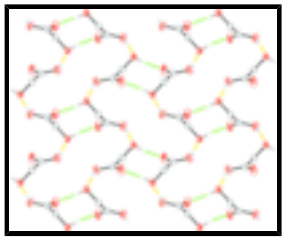

Fig. 3. View of a hydrophilic sheet, projected onto the (100) plane (twice the unit vectors in *b* and *c*). Norbornane-C atoms including their H atoms are omitted except for C2. Hydroxyl-OH···carboxyl-O bonds are drawn in green, the shorter carboxyl-OH···hydroxyl-O bonds are drawn in yellow.

2-Hydroxybicyclo[2.2.1]heptane-2-*endo*-carboxylic acid

Crystal data

$C_8H_{12}O_3$	F_{000} = 336
M_r = 156.18	D_x = 1.339 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 17661 reflections
a = 11.4780 (6) Å	θ = 3.1–27.5º
b = 6.8145 (3) Å	μ = 0.10 mm^{-1}
c = 10.1444 (5) Å	T = 200 (2) K
β = 102.506 (2)º	Platelet, colourless
V = 774.64 (7) Å^3	0.20 × 0.18 × 0.01 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	1314 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	R_{int} = 0.040
Monochromator: MONTEL, graded multilayered X-ray optics	θ_{max} = 27.4º
T = 200(2) K	θ_{min} = 3.5º
thick–slice ω and φ scans	h = −14→14
Absorption correction: none	k = −8→8
6408 measured reflections	l = −13→13
1765 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

$R[F^2 > 2\sigma(F^2)] = 0.045$	Only H-atom displacement parameters refined
$wR(F^2) = 0.119$	$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.3026P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
1765 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
103 parameters	$\Delta\rho_{min} = -0.21$ 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. Comment on the refU data value for H atoms: H atoms were refined as riding on their parent atoms (AFIX 147). One common isotropic displacement parameter for all H atoms was refined to $U_{iso} = 0.050$ (3) Å^2.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	0.38046 (10)	−0.21347 (15)	0.39994 (10)	0.0309 (3)
H2	0.4087	−0.1587	0.4742	0.0485 (16)*
O81	0.53499 (10)	0.0669 (2)	0.34527 (11)	0.0448 (4)
O82	0.43585 (10)	0.06668 (17)	0.13284 (11)	0.0380 (3)
H82	0.4970	0.1269	0.1221	0.0485 (16)*
C1	0.26245 (14)	0.0878 (2)	0.35726 (16)	0.0327 (4)
H1	0.2991	0.1347	0.4504	0.0485 (16)*
C2	0.33717 (13)	−0.0665 (2)	0.29946 (14)	0.0271 (3)
C3	0.24406 (14)	−0.1625 (2)	0.18421 (15)	0.0318 (4)
H31	0.2641	−0.1386	0.0954	0.0485 (16)*
H32	0.2391	−0.3057	0.1983	0.0485 (16)*
C4	0.12712 (15)	−0.0606 (3)	0.19329 (18)	0.0397 (4)
H4	0.0533	−0.1354	0.1518	0.0485 (16)*
C5	0.13144 (17)	0.1498 (3)	0.14017 (19)	0.0469 (5)
H51	0.0523	0.2139	0.1270	0.0485 (16)*
H52	0.1581	0.1514	0.0537	0.0485 (16)*
C6	0.22312 (17)	0.2516 (3)	0.2530 (2)	0.0446 (5)
H61	0.2916	0.3019	0.2183	0.0485 (16)*
H62	0.1861	0.3618	0.2926	0.0485 (16)*
C7	0.14432 (15)	−0.0255 (3)	0.34493 (18)	0.0391 (4)
H71	0.1540	−0.1487	0.3978	0.0485 (16)*
H72	0.0801	0.0557	0.3682	0.0485 (16)*
C8	0.44562 (13)	0.0280 (2)	0.26121 (15)	0.0280 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0344 (6)	0.0338 (6)	0.0236 (5)	0.0030 (5)	0.0044 (5)	0.0023 (4)
O81	0.0329 (7)	0.0722 (9)	0.0275 (6)	−0.0141 (6)	0.0027 (5)	−0.0006 (6)
O82	0.0385 (7)	0.0480 (7)	0.0271 (6)	−0.0141 (5)	0.0060 (5)	0.0024 (5)
C1	0.0329 (9)	0.0341 (8)	0.0320 (8)	0.0027 (7)	0.0090 (7)	−0.0024 (7)
C2	0.0272 (8)	0.0296 (8)	0.0238 (7)	0.0014 (6)	0.0039 (6)	0.0012 (6)
C3	0.0316 (8)	0.0342 (8)	0.0286 (8)	−0.0041 (7)	0.0039 (6)	−0.0023 (6)
C4	0.0264 (8)	0.0508 (11)	0.0397 (10)	−0.0029 (7)	0.0023 (7)	0.0011 (8)
C5	0.0409 (10)	0.0539 (11)	0.0442 (11)	0.0122 (9)	0.0057 (8)	0.0107 (9)
C6	0.0451 (10)	0.0369 (9)	0.0545 (11)	0.0084 (8)	0.0168 (9)	0.0055 (8)
C7	0.0319 (9)	0.0452 (10)	0.0425 (10)	0.0024 (7)	0.0129 (7)	0.0024 (8)
C8	0.0288 (8)	0.0299 (8)	0.0252 (8)	0.0014 (6)	0.0052 (6)	−0.0021 (6)

Geometric parameters (Å, °)

O2—C2	1.4385 (17)	C3—H31	0.990
O2—H2	0.840	C3—H32	0.990
O81—C8	1.2127 (18)	C4—C7	1.527 (2)
O82—C8	1.3094 (19)	C4—C5	1.536 (3)
O82—H82	0.840	C4—H4	1.000
C1—C6	1.537 (2)	C5—C6	1.541 (3)
C1—C7	1.542 (2)	C5—H51	0.990
C1—C2	1.550 (2)	C5—H52	0.990
C1—H1	1.000	C6—H61	0.990
C2—C8	1.525 (2)	C6—H62	0.990
C2—C3	1.548 (2)	C7—H71	0.990
C3—C4	1.532 (2)	C7—H72	0.990
C2—O2—H2	109.5	C3—C4—H4	114.6
C8—O82—H82	109.5	C5—C4—H4	114.6
C6—C1—C7	100.70 (14)	C4—C5—C6	103.19 (14)
C6—C1—C2	109.66 (13)	C4—C5—H51	111.1
C7—C1—C2	100.28 (13)	C6—C5—H51	111.1
C6—C1—H1	114.8	C4—C5—H52	111.1
C7—C1—H1	114.8	C6—C5—H52	111.1
C2—C1—H1	114.8	H51—C5—H52	109.1
O2—C2—C8	107.39 (12)	C1—C6—C5	103.65 (15)
O2—C2—C3	108.50 (12)	C1—C6—H61	111.0
C8—C2—C3	117.04 (12)	C5—C6—H61	111.0
O2—C2—C1	109.80 (12)	C1—C6—H62	111.0
C8—C2—C1	111.01 (12)	C5—C6—H62	111.0
C3—C2—C1	102.96 (12)	H61—C6—H62	109.0
C4—C3—C2	103.53 (12)	C4—C7—C1	94.58 (13)
C4—C3—H31	111.1	C4—C7—H71	112.8
C2—C3—H31	111.1	C1—C7—H71	112.8
C4—C3—H32	111.1	C4—C7—H72	112.8

C2—C3—H32	111.1	C1—C7—H72	112.8
H31—C3—H32	109.0	H71—C7—H72	110.3
C7—C4—C3	102.00 (13)	O81—C8—O82	122.18 (14)
C7—C4—C5	101.54 (15)	O81—C8—C2	121.75 (14)
C3—C4—C5	107.87 (14)	O82—C8—C2	116.06 (13)
C7—C4—H4	114.6		
C6—C1—C2—O2	176.39 (13)	C7—C1—C6—C5	−35.60 (16)
C7—C1—C2—O2	−78.23 (14)	C2—C1—C6—C5	69.49 (17)
C6—C1—C2—C8	57.80 (17)	C4—C5—C6—C1	0.73 (18)
C7—C1—C2—C8	163.18 (12)	C3—C4—C7—C1	55.55 (15)
C6—C1—C2—C3	−68.22 (16)	C5—C4—C7—C1	−55.76 (15)
C7—C1—C2—C3	37.16 (14)	C6—C1—C7—C4	55.89 (15)
O2—C2—C3—C4	113.76 (13)	C2—C1—C7—C4	−56.58 (14)
C8—C2—C3—C4	−124.61 (14)	O2—C2—C8—O81	−40.91 (19)
C1—C2—C3—C4	−2.57 (15)	C3—C2—C8—O81	−163.13 (15)
C2—C3—C4—C7	−33.46 (16)	C1—C2—C8—O81	79.12 (19)
C2—C3—C4—C5	73.00 (16)	O2—C2—C8—O82	139.88 (13)
C7—C4—C5—C6	34.87 (17)	C3—C2—C8—O82	17.66 (19)
C3—C4—C5—C6	−71.91 (17)	C1—C2—C8—O82	−100.09 (15)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O81^{i}	0.84	1.91	2.7447 (16)	172
O82—H82···O2^{ii}	0.84	1.83	2.6653 (15)	173

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

Fig. 1

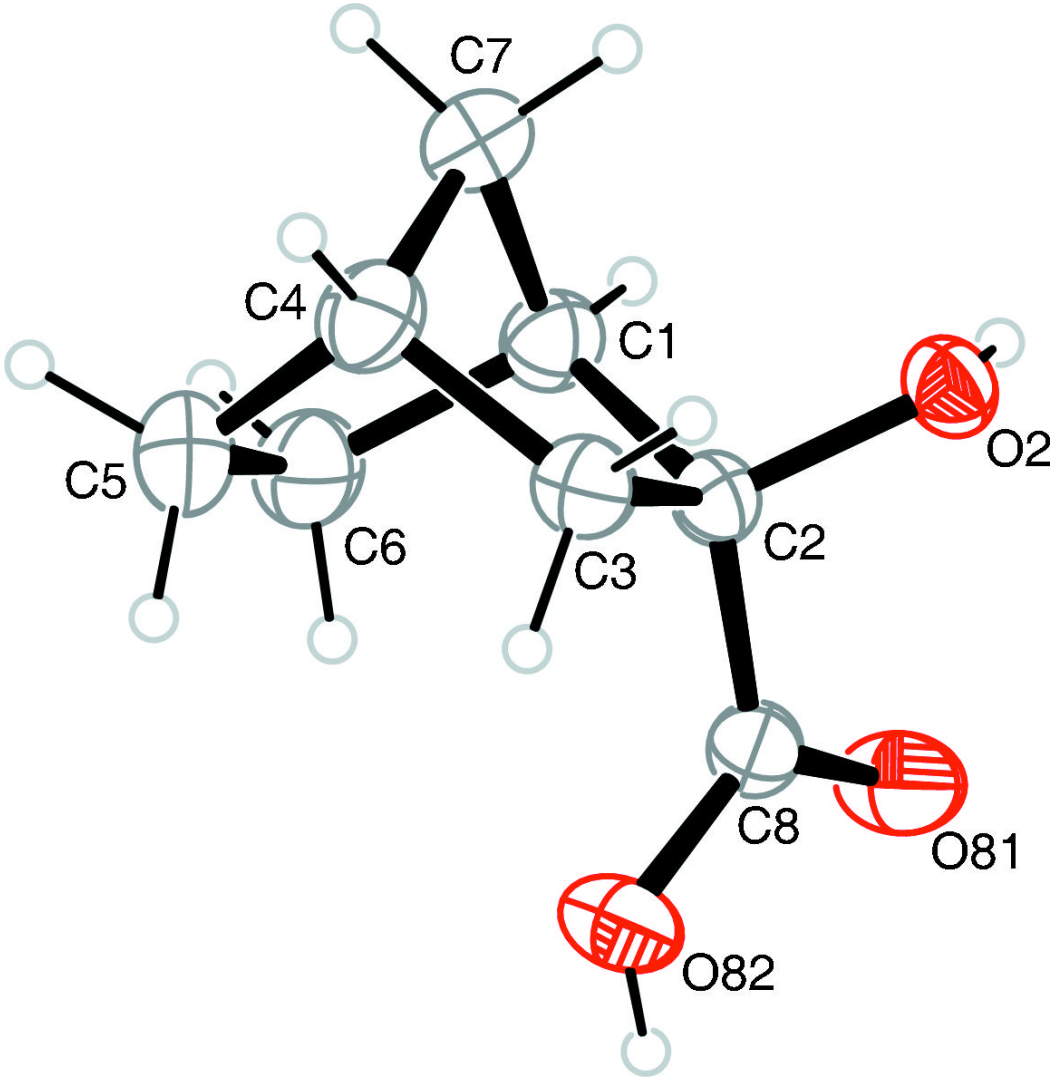

Fig. 2

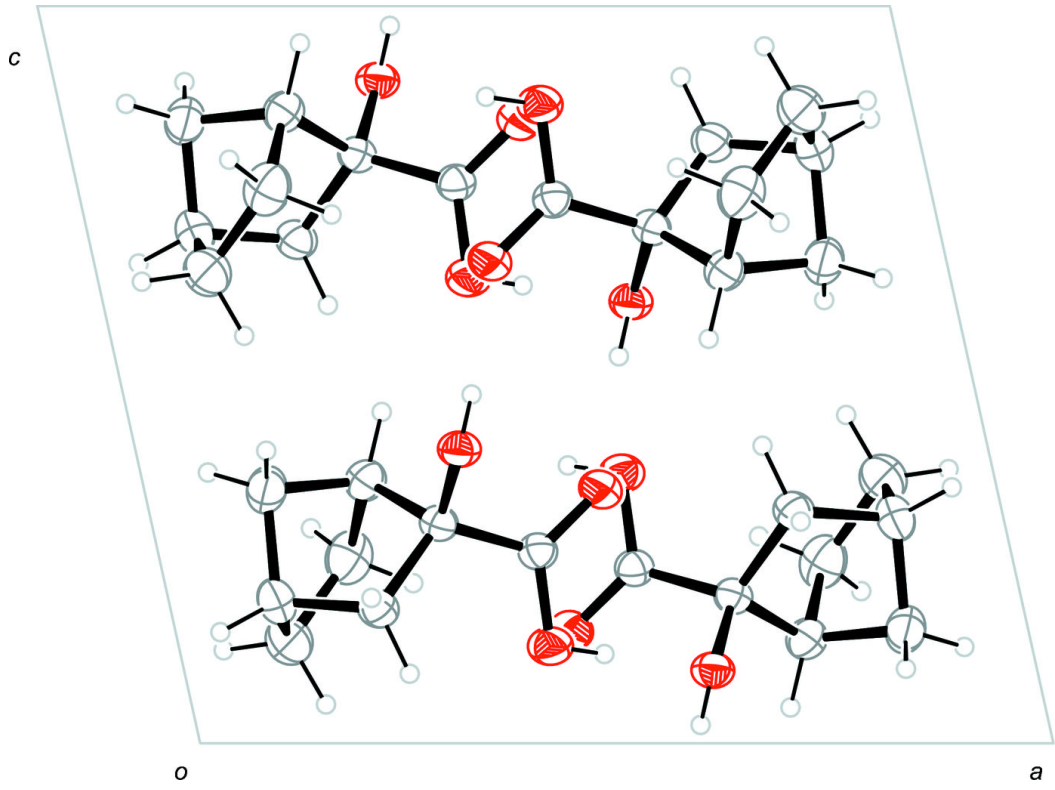

Fig. 3