Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-Hydroxycyclohexanecarboxylic acid

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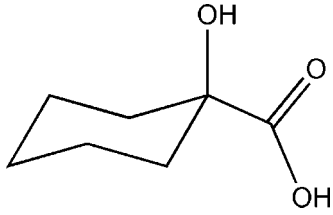
Received 14 August 2007; accepted 20 September 2007

Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.002 Å; R factor = 0.032; wR factor = 0.082; data-to-parameter ratio = 13.0.

The title compound, $C_7H_{12}O_3$, was synthesized as an intermediate for the synthesis of the selective broad-spectrum nonsystemic acaricide spirodiclofen (trade name Envidor). The cyclohexane ring adopts a chair conformation. The molecules pack in layers, with O—H···O hydrogen bonds connecting the layers on one side and only van der Waals interactions on the other side.

Related literature

For the biological activity of the title compound, see: Thomas *et al.* (2003). For a similar structure, see: Abell *et al.* (1988).



Experimental

Crystal data

$C_7H_{12}O_3$
M_r = 144.17
Monoclinic, $P2_1/c$
a = 11.790 (2) Å
b = 6.7956 (14) Å
c = 9.6100 (19) Å
β = 104.97 (3)°
V = 743.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 153 (2) K
0.24 × 0.13 × 0.09 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.917, T_{max} = 0.991
5504 measured reflections
1303 independent reflections
1230 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.082
S = 1.08
1303 reflections
100 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2C···O3^{i}	0.91 (2)	1.74 (2)	2.6221 (12)	161.0 (18)
O3—H3A···O1^{ii}	0.848 (18)	1.885 (18)	2.7285 (12)	173.6 (16)

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

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2157).

References

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 doi:10.1107/S1600536807046351

supplementary materials

Acta Cryst. (2007). E**63**, o4213 [**doi:10.1107/S1600536807046351**]

1-Hydroxycyclohexanecarboxylic acid

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Comment

Spirodiclofen (Trade name: Envidor) is a selective, non-systemic acaricide from the new chemical class of tetronic acid derivatives and was publicly introduced by Bayer CropScience at the BCPC Conference at Brighton in 2000 (Thomas *et al.*, 2003). It is highly active against spider mites and has been developed for citrus, pome fruits, grapes and nuts. Spirodiclofen is a broad-spectrum acaricide with excellent efficacy against all relevant phytophagous mite species such as Panonychus, Tetranychus, Phyllocoptruta, Brevipalpus and Aculus. With an LC50 value of 0.1 p.p.m. and 0.32 p.p.m. for T. urticae and P. ulmi, respectively, this compound is clearly outperforming the older standard acaricides and can compete with the best commercially available acaricides. The title compound (I) is as an intermediate for the synthesis of spirodiclofen.

In (I) (Fig. 1), all bond lengths and angles are normal and in a good agreement with those reported previously (Abell *et al.*, 1988). The cyclohexane ring (C1—C6) adopts a chair conformation. The molecules are in layers with strong intermolecular O—H.·O hydrogen bonds connecting the layers on one side and only van der Waals interactions on the other side. The O–H–O interactions give rise to a hydrogen bonded ten-membered ring.

Experimental

Sodium cyanide (5.5 mmol), was suspended in a solution of cyclohexanone (5.0 mmol) in 20 ml of ethyl ether in a flask equipped with stirrer and reflux condenser. Concentrated hydrochloric acid(5.6 mmol) was slowly added from a dropping-funnel during 30 minutes while maintaining the temperature at 15–20°. Water was then added to dissolve the precipitated sodium chloride. The ether solution of the nitrile was transferred to a separatory-funnel, washed with water, and dried over sodium sulfate, and the ether was removed on a steam-bath. The oil residue was then heated for 4 h on the steam-bath with concentrated hydrochloric acid(5.6 mmol), with strring. Cold water was added to dissolve the ammonium chloride formed. Upon cooling, the acid was then filtered and crystallized from ethyl ether to afford the title compound(0.47 g, yield 95%). Single crystals suitable for X-ray measurement were obtained by recrystallization from petrol ether at room temperature.

Refinement

All H atoms were found on difference maps. The carboxylic acid and hydroxyl H atoms were refined freely, giving an O—H bond distance of 0.91 or 0.85 Å. The remaining H atoms were placed in calculated positions, with C—H = 0.99 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) = 1.2 times U_{eq}(C).

Figures

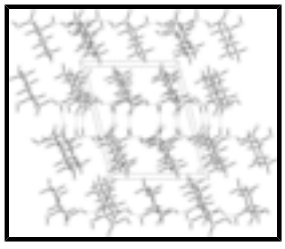

Fig. 1. View of the title compound (I), with displacement ellipsoids drawn at the 35% probability level.

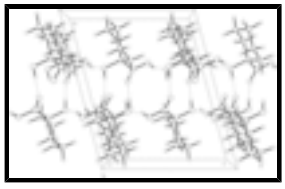

Fig. 2. A packing diagram of the molecule of the title compound, view down *b* axis. Hydrogen bonds are shown as dashed lines.

1-Hydroxycyclohexanecarboxylic acid

Crystal data

$C_7H_{12}O_3$	F_{000} = 312
M_r = 144.17	D_x = 1.287 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 6207 reflections
a = 11.790 (2) Å	θ = 6.0–55.0º
b = 6.7956 (14) Å	μ = 0.10 mm^{-1}
c = 9.6100 (19) Å	T = 153 (2) K
β = 104.97 (3)º	Platelte, colorless
V = 743.8 (3) Å^3	0.24 × 0.13 × 0.09 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer	1303 independent reflections
Radiation source: Rotating Anode	1230 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 153(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 3.5º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	h = −14→13
T_{min} = 0.917, T_{max} = 0.991	k = −7→8
5504 measured reflections	l = −11→11

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.082$

$S = 1.08$

1303 reflections

100 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0381P)^2 + 0.2366P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.30$ e Å^{-3}

$\Delta\rho_{min} = -0.17$ e Å^{-3}

Extinction correction: SHELXTL (Sheldrick, 2001), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.066 (6)

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}
O1	0.55406 (8)	0.26866 (14)	0.84780 (10)	0.0378 (3)
O2	0.68200 (8)	0.19056 (14)	1.05494 (9)	0.0345 (3)
O3	0.64115 (7)	0.00027 (11)	0.69790 (7)	0.0205 (2)
C1	0.69002 (9)	−0.18621 (16)	0.92350 (11)	0.0207 (3)
H1A	0.7201	−0.1693	1.0289	0.025*
H1B	0.6071	−0.2288	0.9036	0.025*
C2	0.76114 (11)	−0.34585 (17)	0.87291 (12)	0.0275 (3)
H2A	0.7238	−0.3777	0.7708	0.033*
H2B	0.7610	−0.4666	0.9305	0.033*
C3	0.88760 (11)	−0.28039 (19)	0.88789 (14)	0.0319 (3)
H3B	0.9280	−0.2644	0.9912	0.038*
H3C	0.9295	−0.3832	0.8477	0.038*
C4	0.89215 (10)	−0.08660 (19)	0.80944 (12)	0.0279 (3)
H4A	0.8585	−0.1058	0.7048	0.033*
H4B	0.9749	−0.0449	0.8248	0.033*

C5	0.82369 (9)	0.07401 (17)	0.86368 (11)	0.0218 (3)
H5A	0.8249	0.1961	0.8078	0.026*
H5B	0.8622	0.1023	0.9661	0.026*
C6	0.69590 (9)	0.01196 (15)	0.84904 (11)	0.0179 (3)
C7	0.63451 (9)	0.17070 (16)	0.91563 (11)	0.0213 (3)
H2C	0.6523 (17)	0.298 (3)	1.090 (2)	0.063 (5)*
H3A	0.5775 (15)	−0.064 (3)	0.6839 (16)	0.041 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0331 (5)	0.0437 (6)	0.0301 (5)	0.0181 (4)	−0.0035 (4)	−0.0052 (4)
O2	0.0483 (6)	0.0328 (5)	0.0179 (4)	0.0178 (4)	0.0004 (4)	−0.0057 (4)
O3	0.0233 (4)	0.0233 (4)	0.0126 (4)	−0.0060 (3)	0.0008 (3)	0.0005 (3)
C1	0.0258 (6)	0.0211 (6)	0.0149 (5)	−0.0019 (4)	0.0048 (4)	0.0016 (4)
C2	0.0427 (7)	0.0193 (6)	0.0212 (6)	0.0014 (5)	0.0096 (5)	−0.0001 (4)
C3	0.0363 (7)	0.0307 (7)	0.0317 (7)	0.0118 (5)	0.0140 (5)	0.0018 (5)
C4	0.0244 (6)	0.0362 (7)	0.0244 (6)	0.0014 (5)	0.0090 (5)	0.0008 (5)
C5	0.0209 (6)	0.0229 (6)	0.0195 (5)	−0.0038 (4)	0.0013 (4)	0.0007 (4)
C6	0.0204 (5)	0.0195 (6)	0.0120 (5)	−0.0010 (4)	0.0010 (4)	−0.0005 (4)
C7	0.0222 (5)	0.0212 (6)	0.0188 (5)	−0.0008 (4)	0.0021 (4)	−0.0003 (4)

Geometric parameters (Å, °)

O1—C7	1.2034 (14)	C2—H2B	0.9900
O2—C7	1.3178 (14)	C3—C4	1.5252 (17)
O2—H2C	0.91 (2)	C3—H3B	0.9900
O3—C6	1.4311 (13)	C3—H3C	0.9900
O3—H3A	0.848 (18)	C4—C5	1.5270 (17)
C1—C2	1.5254 (16)	C4—H4A	0.9900
C1—C6	1.5349 (14)	C4—H4B	0.9900
C1—H1A	0.9900	C5—C6	1.5356 (15)
C1—H1B	0.9900	C5—H5A	0.9900
C2—C3	1.5266 (18)	C5—H5B	0.9900
C2—H2A	0.9900	C6—C7	1.5285 (15)
C7—O2—H2C	110.9 (12)	C3—C4—H4A	109.4
C6—O3—H3A	109.8 (10)	C5—C4—H4A	109.4
C2—C1—C6	112.38 (9)	C3—C4—H4B	109.4
C2—C1—H1A	109.1	C5—C4—H4B	109.4
C6—C1—H1A	109.1	H4A—C4—H4B	108.0
C2—C1—H1B	109.1	C4—C5—C6	111.41 (9)
C6—C1—H1B	109.1	C4—C5—H5A	109.3
H1A—C1—H1B	107.9	C6—C5—H5A	109.3
C1—C2—C3	111.50 (10)	C4—C5—H5B	109.3
C1—C2—H2A	109.3	C6—C5—H5B	109.3
C3—C2—H2A	109.3	H5A—C5—H5B	108.0
C1—C2—H2B	109.3	O3—C6—C7	109.02 (8)
C3—C2—H2B	109.3	O3—C6—C1	111.20 (8)

H2A—C2—H2B	108.0	C7—C6—C1	109.92 (9)
C4—C3—C2	111.30 (10)	O3—C6—C5	106.42 (9)
C4—C3—H3B	109.4	C7—C6—C5	109.11 (9)
C2—C3—H3B	109.4	C1—C6—C5	111.08 (9)
C4—C3—H3C	109.4	O1—C7—O2	124.20 (11)
C2—C3—H3C	109.4	O1—C7—C6	123.77 (10)
H3B—C3—H3C	108.0	O2—C7—C6	112.01 (9)
C3—C4—C5	111.05 (9)		
C6—C1—C2—C3	53.49 (12)	C4—C5—C6—C7	175.56 (9)
C1—C2—C3—C4	−55.00 (13)	C4—C5—C6—C1	54.23 (11)
C2—C3—C4—C5	56.54 (13)	O3—C6—C7—O1	−0.75 (15)
C3—C4—C5—C6	−56.31 (12)	C1—C6—C7—O1	−122.88 (12)
C2—C1—C6—O3	65.34 (11)	C5—C6—C7—O1	115.09 (12)
C2—C1—C6—C7	−173.83 (9)	O3—C6—C7—O2	−179.18 (9)
C2—C1—C6—C5	−52.98 (12)	C1—C6—C7—O2	58.69 (12)
C4—C5—C6—O3	−66.94 (11)	C5—C6—C7—O2	−63.34 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2C···O3^{i}	0.91 (2)	1.74 (2)	2.6221 (12)	161.0 (18)
O3—H3A···O1^{ii}	0.848 (18)	1.885 (18)	2.7285 (12)	173.6 (16)

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

Fig. 1

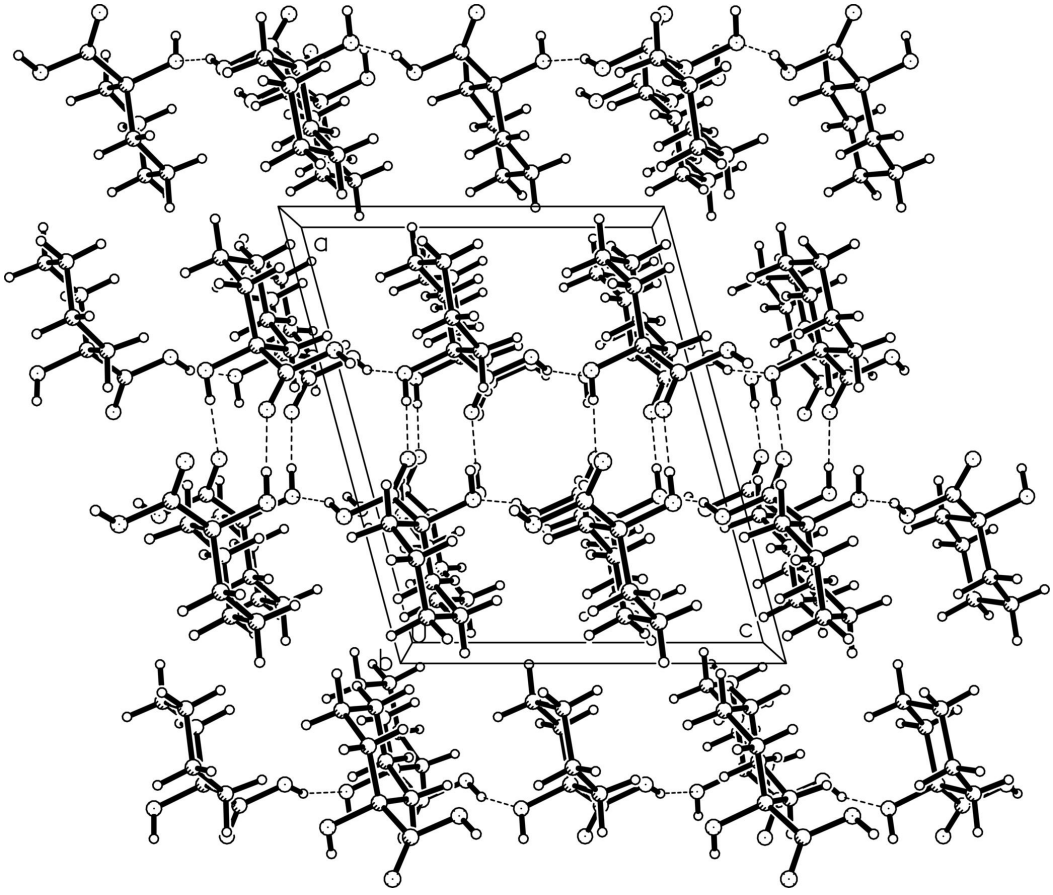

Fig. 2