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(R*,S*)-Methyl 2-(2′-methylenecyclopropyl)-hydroxyacetate

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The relative configuration of the title molecule, $C_7H_{10}O_3$, has been determined. The asymmetric C atoms are of opposite chirality (R,S or S,R), as opposed to having the same chirality (R,R or S,S). The molecule adopts an antiperiplanar conformation around the bond connecting the cyclopropyl and carbonyl groups. In the crystal structure, molecules form hydrogen-bonded chains.

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Key indicators

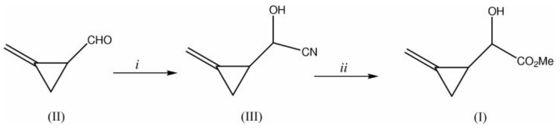
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.001 Å
R factor = 0.035
wR factor = 0.104
Data-to-parameter ratio = 15.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

For the stereochemical assignment of (2′-methylenecyclopropyl)carbinols see Maurin & Bertrand (1970) and Lautens & Delanghe (1994).

The title compound (Bagutski, 2003), (I), was prepared by methanolysis of the cyanohydrine, (III), obtained from the known methylenecyclopropanecarboxaldehyde, (II) (Le Corre *et al.*, 1994), as a key intermediate along the route to new vinylcyclopropane monomers for radical ring-opening polymerization (Moszner *et al.*, 2003; de Meijere *et al.*, 2004). The crude mixture of diastereomers was separated by column chromatography and one of the two diastereomers gave good quality crystals. [In the scheme below: (i) NaCN, NH_4Cl, Et_2O, H_2O, 283 K, 40 min; (ii) 1: HCl, MeOH, Et_2O, 275 K, 12 h; 2: H_2O, 273 K, 30 min.]



The structure of the crystalline diastereomer, (I), has been determined by X-ray crystallography and we report here the results of this study.

The molecular structure and the atom-labelling scheme are shown in Fig. 1. The geometrical parameters of the molecule are close to those found in similar compounds (*e.g.* Delanghe *et al.*, 1995). The molecule adopts an *anti* conformation around the C2—C5 bond, with a dihedral angle H2—C2—C5—H5 of −173 (1)°. In the crystal structure, the molecules of (R*,S*)-(I) form hydrogen-bonded chains (Fig. 2) along the b axis (Fig. 3). In addition to the strong O1—H⋯O1^{i} [symmetry code: (i) $-x, y-\frac{1}{2}, \frac{1}{2}-z$] hydrogen bond there is a relatively short contact O1—H⋯O2^{i} [O⋯O = 3.03 (2) Å and angle = 131 (1)°], which may be regarded as the minor component of a bifurcated hydrogen bond according to Steiner (2002).

Experimental

The title compound was prepared by treatment of (2-methylenecyclopropane)carboxaldehyde, (II), with NaCN/NH_4Cl in a mixture of water and diethyl ether (1:1), subsequent methanolysis of the

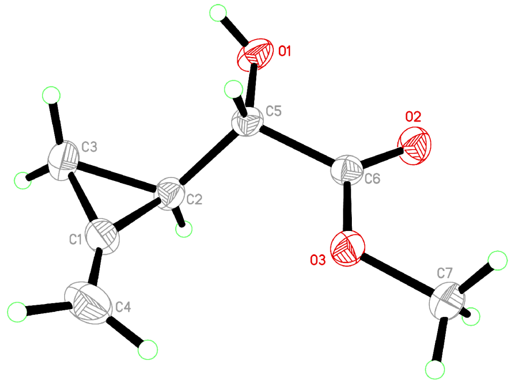


Figure 1
The molecular structure of (I) and the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level.

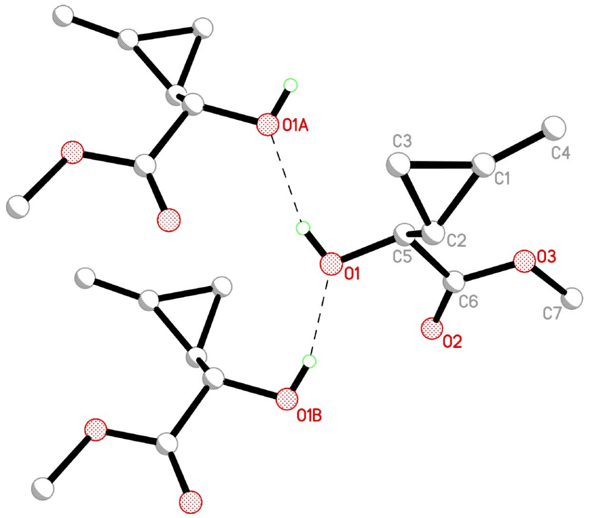


Figure 2
Hydrogen-bonded chains in the structure of (I).

crude cyanohydrin with HCl/MeOH and hydrolysis of the resulting iminoester hydrochloride. The crude reaction mixture was subjected to column chromatography on silica gel, eluting with pentane/diethyl ether. Single crystals of (*R**,*S**)-(I) suitable for X-ray diffraction were grown by slow evaporation of an octane–diethyl ether solution at ambient temperature.

Crystal data

$C_7H_{10}O_3$
$M_r = 142.15$
Monoclinic, $P2_1/c$
$a = 7.2608$ (2) Å
$b = 5.0643$ (2) Å
$c = 19.9397$ (6) Å
$\beta = 92.89$ (1)°
$V = 732.27$ (4) Å^3
$Z = 4$

$D_x = 1.289$ Mg m^{-3}
Mo $K\alpha$
Cell parameters from 4735 reflections
$\theta = 2.8$–30.9°
$\mu = 0.10$ mm^{-1}
$T = 120$ (2) K
Plate, colourless
0.64 × 0.22 × 0.12 mm

Data collection

Bruker SMART 6000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.837$, $T_{\max} = 0.988$
7561 measured reflections

2017 independent reflections
1830 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.017$
$\theta_{\max} = 29.5°$
$h = -10 \rightarrow 9$
$k = -6 \rightarrow 7$
$l = -27 \rightarrow 27$

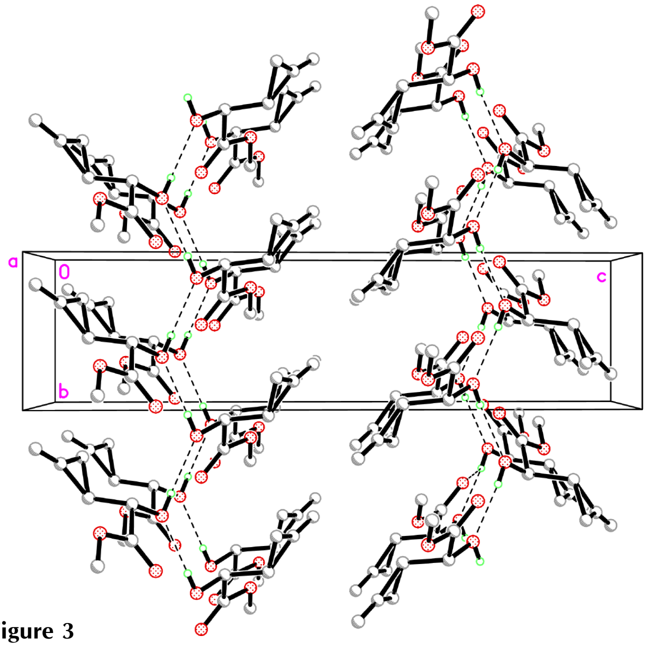


Figure 3
Packing of the molecules in the crystal, viewed along the *b* axis. H atoms have been omitted for clarity.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.104$
$S = 1.03$
2017 reflections
131 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.1P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.33$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C5	1.4133 (9)	C1—C3	1.4653 (13)
O2—C6	1.2039 (10)	C1—C2	1.4651 (12)
O3—C6	1.3311 (9)	C2—C5	1.5157 (10)
O3—C7	1.4473 (10)	C2—C3	1.5391 (11)
C1—C4	1.3130 (14)	C5—C6	1.5214 (10)
C6—O3—C7	115.29 (7)	C1—C3—C2	58.31 (6)
C4—C1—C3	149.20 (9)	O1—C5—C2	111.54 (6)
C4—C1—C2	147.23 (9)	O1—C5—C6	107.19 (6)
C3—C1—C2	63.37 (6)	C2—C5—C6	111.61 (6)
C1—C2—C5	119.26 (7)	O2—C6—O3	124.93 (7)
C1—C2—C3	58.32 (6)	O2—C6—C5	124.42 (7)
C5—C2—C3	116.68 (7)	O3—C6—C5	110.64 (6)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1O$\cdots$O1$^{\rm i}$	0.819 (17)	2.027 (16)	2.7873 (5)	154.1 (15)

Symmetry code: (i) $-x, y-\frac{1}{2}, \frac{1}{2}-z$.

All H atoms were located in difference Fourier maps and included in the refinement with isotropic displacement parameters. The C—H distances range from 0.95 (1) to 0.98 (1) Å and the O—H distance is 0.82 (2) Å.

Data collection: *SMART* (Bruker, 1998–2000); cell refinement: *SAINT* (Bruker, 1998–2000); data reduction: *SAINT*; program(s)

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used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998–2000); software used to prepare material for publication: *SHELXTL*.

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References

Bagutski, V. (2003). Part of a forthcoming dissertation, University of Göttingen, Germany.

Bruker (1998–2000). *SMART-NT* (Version 5.0), *SAINT-NT* (Version 5.0) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.

Delanghe, P. H. M., Lautens, M. & Lough, A. J. (1995). *Acta Cryst.* C**51**, 700–703.

Lautens, M. & Delanghe, P. H. M. (1994). *J. Am. Chem. Soc.* **116**, 8526–8535.

Le Corre, M., Hercouet, A. & Bessieres, B. (1994). *J. Org. Chem.* **59**, 5483–5484.

Maurin, R. & Bertrand, M. (1970). *Bull. Chim. Soc. Fr.* pp. 2261–2270.

Meijere, A. de, Bagutski, V., Zeuner, F., Fischer, U. K., Rheinberger, V. & Moszner, N. (2004). *Macromol. Chem. Phys.* Submitted.

Moszner, N., Zeuner, F., Fischer, U. K., Rheinberger, V., de Meijere, A. & Bagutski, V. (2003). *Macromol. Rapid Commun.* **24**, 269–271.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Steiner, T. (2002). *Angew. Chem. Int. Ed.* **41**, 48–76.

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