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(−)-(1*S*,5*R*)-2-Oxabicyclo[3.3.1]nonan-3-one

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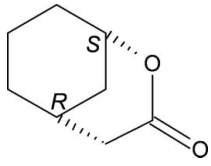
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.043; wR factor = 0.096; data-to-parameter ratio = 10.2.

In the title compound, $C_8H_{12}O_2$, the cyclohexane ring exhibits a chair conformation and the δ-lactone ring is axially bonded to the cyclohexane ring. In the crystal, molecules are linked by C—H···O hydrogen bonds, resulting in ribbons extending along [010].

Related literature

For the synthesis and confirmation of the absolute configuration of the title compound, see Olejniczak (2010); Waschołowski *et al.* (2008); Tzvetkov *et al.* (2006); Xu *et al.* (2002). For related structures see: Yokoyama *et al.* (2003); Schmidt *et al.* (1998); Finet *et al.* (2007); Militsina *et al.* (2005).



Experimental

Crystal data

$C_8H_{12}O_2$
M_r = 140.18
Orthorhombic, $P2_12_12_1$
a = 6.793 (2) Å
b = 7.467 (2) Å
c = 14.170 (4) Å
V = 718.7 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.30 × 0.14 × 0.10 mm

Data collection

Kuma KM-4-CCD diffractometer
4925 measured reflections
935 independent reflections
685 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.096
S = 1.04
935 reflections
92 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O2^{i}	1.00	2.58	3.224 (3)	122

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* (Bruker, 1999); 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: HG2667).

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supporting information

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(−)-(1*S*,5*R*)-2-Oxabicyclo[3.3.1]nonan-3-one

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S1. Comment

The titled compound, $C_8H_{12}O_2$, was prepared in a three step synthesis (Fig. 2). Racemic diethyl 2-(3-oxocyclohexyl)-malonate (**1**) was synthesized as a product of Michael addition of diethyl malonate to cyclohex-2-en-1-one. (-)-Diethyl 2-((*S*)-3-oxocyclohexyl)malonate (**(-)-1**) (ee=98%) and (+)-diethyl 2-((1*R*, 3*R*)-3-hydroxycyclohexyl)malonate (**(+)-2**) (ee=99%) were isolated by micriobial bioreduction using *Absidia coerulea* AM 93. Hydroxydiester-**(+)-2** was subjected to chemical lactonization, leading to (-)-(1*S*, 5*R*)-2-oxabicyclo[3.3.1]nonan-3-one (**(-)-3**) [for more details see Olejniczak, 2010].

The molecular structure of the title compound is shown in Fig. 1. Bond lengths and angles in **(-)-3** are similar to those observed in related structures [Yokoyama *et al.*, 2003; Schmidt *et al.*, 1998; Finet *et al.*, 2007]. As in these related structures, in **(-)-3** the cyclohexane ring reveals *chair* conformation (Fig. 1) and the *δ*-lactone ring is axially bonded to the cyclohexane ring.

It is worth mentioning that the conformation of the *δ*-lactone ring differs a little from those observed in the related structures. According to the numbering scheme employed in this paper, the torsion angles C1 O2 C3 C4 and O2 C3 C4 C5 in the related structures are in the range -7.1 - 0.4 ° and 0.0 - 8.8 °, respectively, and in **(-)-3** values of suitable torsion angles are equal to -18.1 (3) and 23.3 (4) °. However, the values of the torsion angles are similar to those, -17.5 and 25.5 °, observed in one of crystallographically unrelated molecules of 3,9,12a-trimethyl-5-oxotetradecahydro-3,6a- methano-naphtho[2,1-d]oxocine-9-carboxylic acid, in which *δ*-lactone ring axially bonded to cyclohexane ring is observed [Militsina *et al.*, 2005].

The structure of **(-)-3** is stabilized by weak intermolecular C—H···O hydrogen bonds and van der Waals contacts. Molecules of **(-)-3** are linked by the C1—H1···O2(2-x, 0.5+y, 0.5-z) hydrogen bonds, resulting in ribbons extended along the [010] direction (Table 1, Fig. 3).

S2. Experimental

Crystals suitable for X-ray structure analysis were obtained directly after purification by column chromatography by slow evaporation of the eluent (petroleum ether : aceton : *iso*-propanol : ethyl acetate (40:1:3:1) v/v) at room temperature.

S3. Refinement

All H atoms were placed at calculated positions and were treated as riding atoms, with C—H distances of 0.99 - 1.00 Å. The absolute configuration of **(-)-3** was choosen on the basis of known absolute configuration of particular substrates: The absolute configuration of **(-)-1** was confirmed by comparison of its optical rotation with the literature data [Wascholowski *et al.*, 2008; Tzvetkov *et al.*, 2006; Xu *et al.*, 2002]. The absolute configuration of the carbon atom bearing hydroxyl group in product **(+)-2** was determined using the Mosher's ester [Olejniczak, 2010].

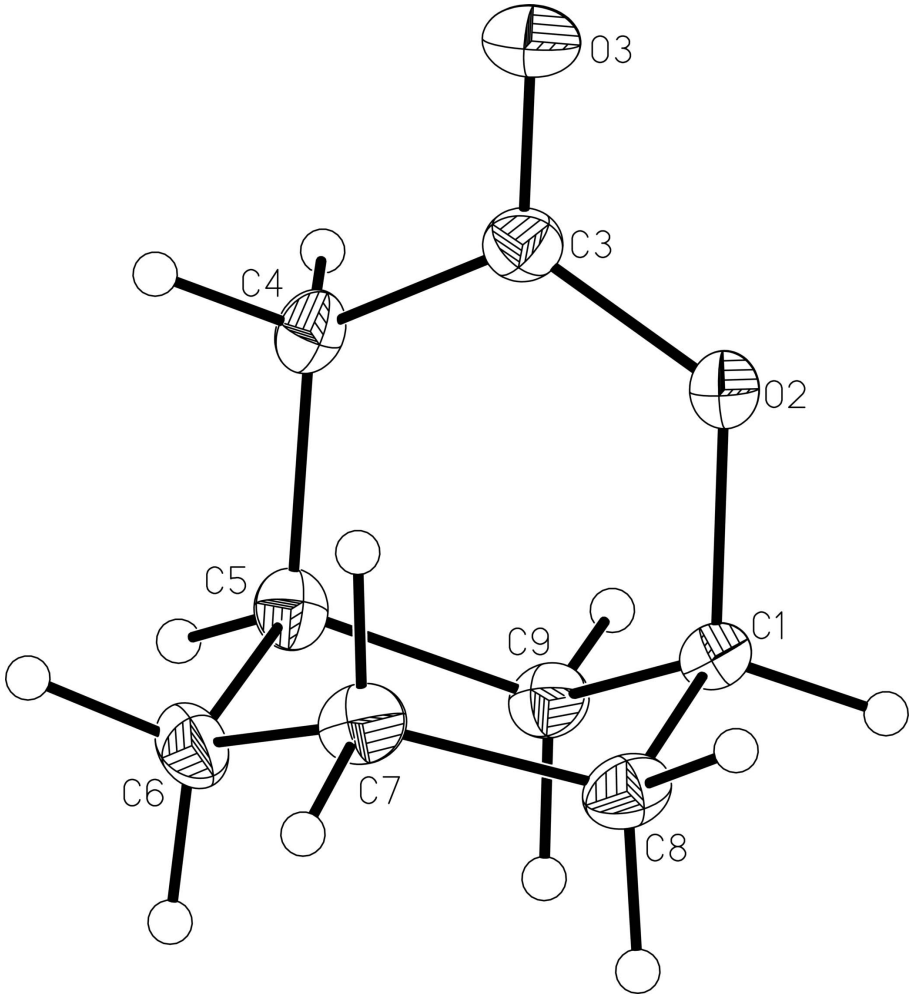

Figure 1

Selected view of **(-)-3** (30% probability thermal ellipsoids).

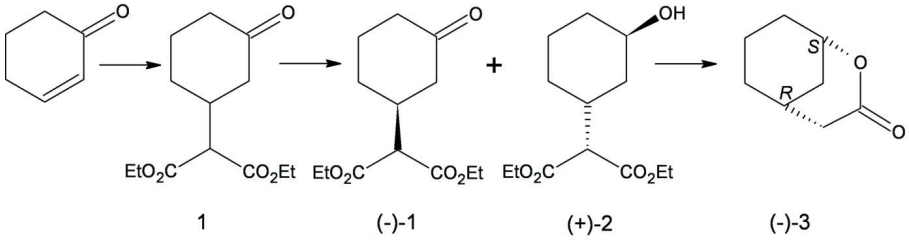

Figure 2

Scheme of a three step synthesis of **(-)-3**.

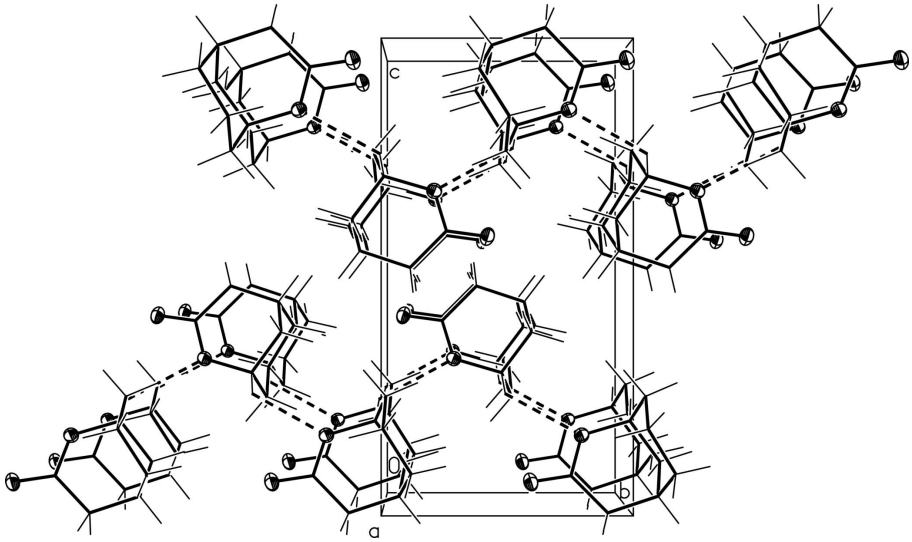

Figure 3

Packing of **(-)-3**.

(-)-(1*S*,5*R*)-2-Oxabicyclo[3.3.1]nonan-3-one

Crystal data

$C_8H_{12}O_2$
$M_r = 140.18$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 6.793$ (2) Å
$b = 7.467$ (2) Å
$c = 14.170$ (4) Å
$V = 718.7$ (4) Å^3
$Z = 4$
$F(000) = 304$
$D_x = 1.295$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1754 reflections
$\theta = 3.0$–$28.8°$
$\mu = 0.09$ mm^{-1}
$T = 100$ K
Needle, colorless
0.30 × 0.14 × 0.10 mm

Data collection

Kuma KM-4-CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
4925 measured reflections
935 independent reflections
685 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.0°$, $\theta_{min} = 3.1°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -15 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.096$
$S = 1.04$
935 reflections
92 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0507P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.004$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.9078 (4)	0.4787 (3)	0.29810 (17)	0.0293 (6)
H1	1.0256	0.4916	0.2564	0.035*
O2	0.8893 (3)	0.2882 (2)	0.32719 (12)	0.0331 (5)
C3	0.7920 (4)	0.2414 (4)	0.40668 (17)	0.0302 (6)
O3	0.7539 (3)	0.0838 (2)	0.41770 (13)	0.0389 (5)
C4	0.7539 (4)	0.3815 (3)	0.48057 (18)	0.0300 (6)
H4A	0.8553	0.3693	0.5304	0.036*
H4B	0.6247	0.3561	0.5100	0.036*
C5	0.7535 (4)	0.5754 (3)	0.44672 (17)	0.0287 (6)
H5	0.7674	0.6561	0.5027	0.034*
C6	0.5660 (4)	0.6256 (4)	0.39356 (19)	0.0347 (7)
H6B	0.4509	0.6041	0.4349	0.042*
H6A	0.5695	0.7549	0.3782	0.042*
C7	0.5413 (4)	0.5192 (4)	0.30310 (17)	0.0344 (7)
H7B	0.4290	0.5683	0.2669	0.041*
H7A	0.5108	0.3931	0.3189	0.041*
C8	0.7258 (4)	0.5254 (4)	0.24230 (17)	0.0334 (6)
H8B	0.7115	0.4402	0.1892	0.040*
H8A	0.7407	0.6471	0.2155	0.040*
C9	0.9334 (4)	0.5985 (4)	0.38359 (19)	0.0302 (7)
H9B	0.9457	0.7250	0.3635	0.036*
H9A	1.0542	0.5648	0.4184	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0333 (15)	0.0256 (14)	0.0290 (14)	0.0004 (12)	0.0060 (13)	0.0044 (12)
O2	0.0360 (11)	0.0311 (10)	0.0322 (10)	0.0031 (8)	0.0082 (9)	−0.0003 (9)
C3	0.0285 (15)	0.0334 (16)	0.0286 (14)	0.0022 (12)	−0.0040 (12)	0.0043 (12)
O3	0.0434 (11)	0.0284 (10)	0.0451 (11)	−0.0025 (10)	−0.0047 (11)	0.0059 (9)
C4	0.0245 (14)	0.0393 (15)	0.0261 (12)	−0.0021 (14)	0.0014 (13)	0.0003 (11)
C5	0.0291 (14)	0.0297 (14)	0.0273 (13)	−0.0034 (13)	−0.0009 (13)	−0.0068 (11)
C6	0.0268 (15)	0.0336 (16)	0.0435 (17)	0.0048 (12)	0.0036 (13)	−0.0038 (13)
C7	0.0284 (14)	0.0368 (16)	0.0381 (17)	−0.0040 (12)	−0.0047 (12)	0.0040 (14)
C8	0.0429 (17)	0.0314 (14)	0.0259 (13)	−0.0055 (14)	−0.0033 (13)	0.0012 (11)
C9	0.0242 (14)	0.0312 (15)	0.0353 (16)	−0.0039 (12)	−0.0012 (12)	0.0037 (12)

Geometric parameters (Å, º)

C1—O2	1.486 (3)	C5—H5	1.0000
C1—C8	1.509 (3)	C6—C7	1.517 (4)
C1—C9	1.516 (4)	C6—H6B	0.9900
C1—H1	1.0000	C6—H6A	0.9900
O2—C3	1.352 (3)	C7—C8	1.522 (3)
C3—O3	1.215 (3)	C7—H7B	0.9900
C3—C4	1.502 (4)	C7—H7A	0.9900
C4—C5	1.526 (3)	C8—H8B	0.9900
C4—H4A	0.9900	C8—H8A	0.9900
C4—H4B	0.9900	C9—H9B	0.9900
C5—C9	1.524 (3)	C9—H9A	0.9900
C5—C6	1.526 (4)		
O2—C1—C8	107.3 (2)	C7—C6—H6B	109.1
O2—C1—C9	110.7 (2)	C5—C6—H6B	109.1
C8—C1—C9	112.1 (2)	C7—C6—H6A	109.1
O2—C1—H1	108.9	C5—C6—H6A	109.1
C8—C1—H1	108.9	H6B—C6—H6A	107.9
C9—C1—H1	108.9	C6—C7—C8	111.8 (2)
C3—O2—C1	121.32 (19)	C6—C7—H7B	109.3
O3—C3—O2	117.5 (2)	C8—C7—H7B	109.3
O3—C3—C4	123.2 (2)	C6—C7—H7A	109.3
O2—C3—C4	119.0 (2)	C8—C7—H7A	109.3
C3—C4—C5	116.2 (2)	H7B—C7—H7A	107.9
C3—C4—H4A	108.2	C1—C8—C7	111.8 (2)
C5—C4—H4A	108.2	C1—C8—H8B	109.3
C3—C4—H4B	108.2	C7—C8—H8B	109.3
C5—C4—H4B	108.2	C1—C8—H8A	109.3
H4A—C4—H4B	107.4	C7—C8—H8A	109.3
C9—C5—C4	106.9 (2)	H8B—C8—H8A	107.9
C9—C5—C6	110.59 (19)	C1—C9—C5	108.1 (2)
C4—C5—C6	112.9 (2)	C1—C9—H9B	110.1
C9—C5—H5	108.8	C5—C9—H9B	110.1
C4—C5—H5	108.8	C1—C9—H9A	110.1
C6—C5—H5	108.8	C5—C9—H9A	110.1
C7—C6—C5	112.4 (2)	H9B—C9—H9A	108.4
C8—C1—O2—C3	−85.9 (3)	C4—C5—C6—C7	−63.5 (3)
C9—C1—O2—C3	36.8 (3)	C5—C6—C7—C8	−50.9 (3)
C1—O2—C3—O3	167.8 (2)	O2—C1—C8—C7	65.6 (3)
C1—O2—C3—C4	−18.1 (3)	C9—C1—C8—C7	−56.1 (3)
O3—C3—C4—C5	−162.9 (3)	C6—C7—C8—C1	50.3 (3)
O2—C3—C4—C5	23.3 (4)	O2—C1—C9—C5	−59.8 (3)
C3—C4—C5—C9	−46.0 (3)	C8—C1—C9—C5	60.0 (3)
C3—C4—C5—C6	75.9 (3)	C4—C5—C9—C1	64.0 (2)
C9—C5—C6—C7	56.2 (3)	C6—C5—C9—C1	−59.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O2^{i}	1.00	2.58	3.224 (3)	122

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