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5,5-Bis(hydroxymethyl)-3-methylcyclohex-2-enone

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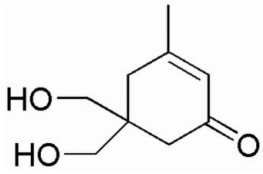
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.055; wR factor = 0.195; data-to-parameter ratio = 18.7.

In the title compound, $C_9H_{14}O_3$, the cyclohexenone ring has an envelope conformation; the flap atom (with the hydroxymethyl groups attached) is displaced by 0.582 (4) Å from the plane of the other five ring atoms. The crystal structure contains an intermolecular O—H···O hydrogen-bonded ring.

Related literature

For related literature, see: Aghil *et al.* (1992); Hu *et al.* (2003); Li & Strobel (2001); Luu *et al.* (2004).



Experimental

Crystal data

$C_9H_{14}O_3$
M_r = 170.21
Triclinic, $P\overline{1}$
a = 5.9791 (3) Å
b = 6.2251 (1) Å
c = 13.7493 (8) Å
α = 90.8104 (17)°
β = 91.3285 (12)°
γ = 117.0728 (15)°
V = 455.38 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 (1) K
0.43 × 0.40 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.958, T_{max} = 0.982
4514 measured reflections
2060 independent reflections
1432 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.195
S = 1.01
2060 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H201···O3^{i}	0.92	1.85	2.738 (2)	163
O3—H301···O2^{ii}	0.95	1.84	2.733 (2)	155

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004) and Larson (1970); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2128).

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supporting information

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5,5-Bis(hydroxymethyl)-3-methylcyclohex-2-enone

Dongmei Cui, Qian Wang, Chen Zhang and Jianming Gu

S1. Comment

Functionalized cyclohex-2-enone derivatives can be used as precursors in the syntheses of some complex compounds, such as vitamin E, amino acids, terpenes *etc*. (Hu *et al.*, 2003). In addition, cyclohex-2-enone derivatives have been shown to have a wide range of biological activities such as antimicrobial (Li *et al.*, 2001) and anticancer (Aghil *et al.*, 1992) activities, and are involved in the protection of cerebral neurocytes (Luu *et al.*, 2004). We are interested in their further pharmaceutical activity.

In this paper, we present an X-ray crystallographic analysis of the title compound (I) (Fig. 1). The cyclohexenone ring has an envelope conformation, such that the plane which is composed of atoms C1, C2 and C6 (forming the flap) and the C2, C3, C4, C5, C6 plane form a dihedral angle of 41.80 (4)°. Two molecules are linked together through O—H···O interactions. Since each molecule contains a hydrogen-bond donor group (–OH) at one end and an acceptor (–OH) at the other, a ring of four H-bonds is formed between these two molecules and a neighboring pair in the crystal lattice (Fig. 2).

S2. Experimental

A solution of 4,4-bis(hydroxymethyl)-2,6-heptanedione(188 mg, 1 mmol) and sodium methoxide (54 mg, 1 mmol) in methanol (10 ml) was heated at 323 K for 4 h. The reaction mixture was acidified with dilute aqueous HCl, then concentrated and partitioned between water and dichloromethane. The pure product was obtained through silica gel chromatography (eluant petroleum ether/ethyl acetate, 1:1), and diffraction quality crystals were obtained by slow evaporation of a dichloromethane / petroleum ether (1:3) solution at room temperature.

S3. Refinement

All H atoms were placed in calculated positions, with C—H distances in the range 0.93–0.98Å and included in the final cycles of refinement in the riding-model approximation, with U_{iso}(H) = 1.2Ueq(C).

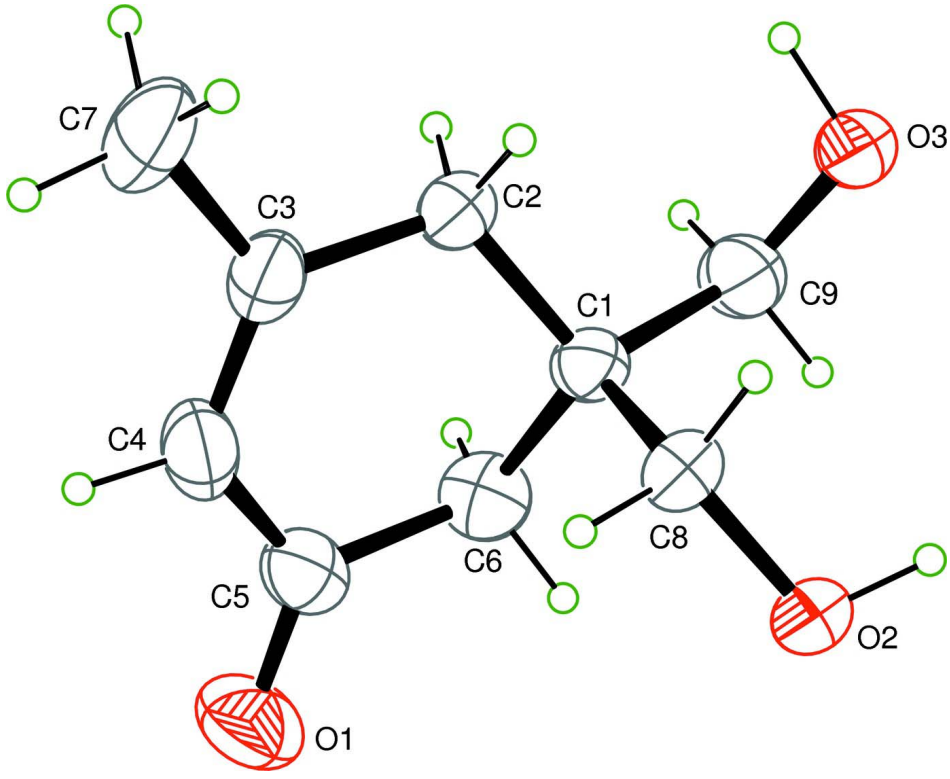

Figure 1

The unit of (I) with atom labels, showing 50% probability displacement ellipsoids.

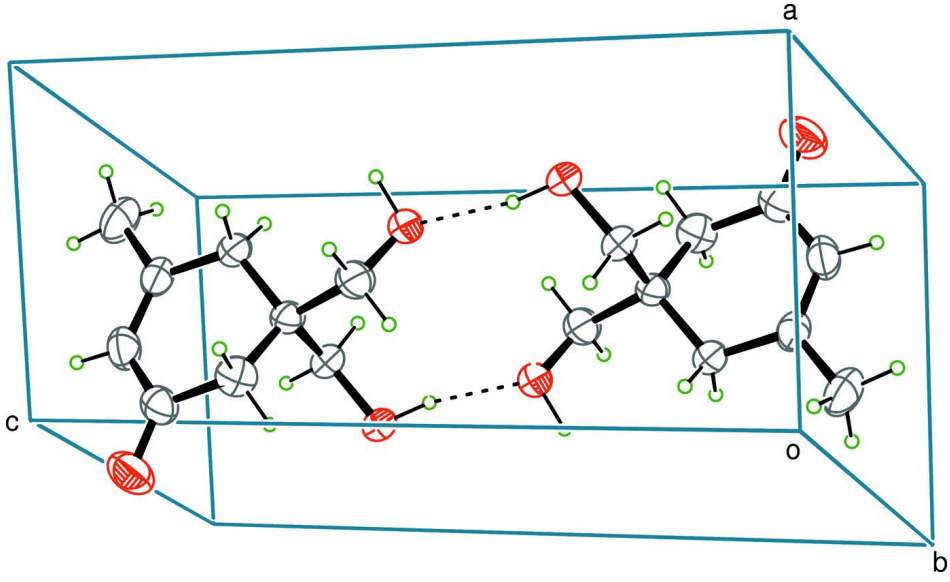

Figure 2

A partial packing diagram viewed along the *b* axis. Hydrogen bonds are drawn as dashed lines.

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Crystal data

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$\alpha = 90.8104$ (17)°
$\beta = 91.3285$ (12)°
$\gamma = 117.0728$ (15)°
$V = 455.38$ (4) Å^3
$Z = 2$
$F(000) = 184.00$
$D_x = 1.241$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Cell parameters from 3491 reflections
$\theta = 3.7$–27.4°
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Chunk, colorless
0.43 × 0.40 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Detector resolution: 10.00 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.958$, $T_{max} = 0.982$
4514 measured reflections
2060 independent reflections
1432 reflections with $F^2 > 2\sigma(F^2)$
$R_{int} = 0.018$
$\theta_{max} = 27.5$°
$h = -7 \rightarrow 7$
$k = -8 \rightarrow 8$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.195$
$S = 1.01$
2060 reflections
110 parameters
H-atom parameters constrained
$w = 1/[0.0027F_o^2 + 5\sigma(F_o^2) + 1]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 107 (30)

Special details

Refinement. Refinement using all reflections. The weighted *R*-factor (*wR*) and goodness of fit (*S*) are based on F^2. *R*-factor (gt) are based on *F*. The threshold expression of $F^2 > 2.0\ \sigma(F^2)$ is used only for calculating *R*-factor (gt).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.1756 (3)	1.1069 (3)	0.16817 (14)	0.0617 (6)
O2	0.8610 (2)	0.5583 (3)	0.38179 (12)	0.0518 (5)
O3	0.3183 (3)	0.5928 (4)	0.43726 (12)	0.0625 (6)
C1	0.6300 (3)	0.7646 (4)	0.31049 (14)	0.0342 (5)
C2	0.4284 (4)	0.7285 (4)	0.23242 (14)	0.0382 (6)
C3	0.5141 (4)	0.7644 (4)	0.13114 (16)	0.0395 (6)
C4	0.7599 (4)	0.8911 (4)	0.11129 (17)	0.0459 (6)
C5	0.9546 (4)	1.0036 (4)	0.18614 (18)	0.0418 (6)
C6	0.8686 (4)	0.9901 (4)	0.29092 (17)	0.0435 (6)
C7	0.3125 (5)	0.6558 (5)	0.05324 (18)	0.0575 (8)
C8	0.6772 (4)	0.5422 (4)	0.31014 (16)	0.0391 (6)
C9	0.5370 (4)	0.7935 (5)	0.41112 (17)	0.0497 (7)

H4	0.8057	0.9066	0.0466	0.055*
H21	0.2949	0.5649	0.2365	0.046*
H22	0.3628	0.8418	0.2467	0.046*
H61	1.0013	0.9953	0.3343	0.052*
H62	0.8404	1.1291	0.3044	0.052*
H71	0.3875	0.6827	−0.0093	0.069*
H72	0.2024	0.7294	0.0564	0.069*
H73	0.2183	0.4855	0.0626	0.069*
H81	0.5204	0.4012	0.3225	0.047*
H82	0.7344	0.5243	0.2465	0.047*
H91	0.6684	0.8209	0.4596	0.060*
H92	0.5042	0.9324	0.4103	0.060*
H201	0.7736	0.4867	0.4357	0.067*
H301	0.1888	0.6149	0.4049	0.081*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0437 (10)	0.0582 (12)	0.0750 (14)	0.0149 (8)	0.0195 (9)	0.0154 (10)
O2	0.0403 (9)	0.0843 (13)	0.0433 (9)	0.0383 (9)	0.0072 (7)	0.0232 (8)
O3	0.0444 (9)	0.1166 (18)	0.0398 (9)	0.0471 (11)	0.0123 (7)	0.0284 (10)
C1	0.0325 (10)	0.0441 (13)	0.0296 (10)	0.0205 (9)	0.0006 (8)	−0.0006 (8)
C2	0.0357 (10)	0.0514 (14)	0.0324 (11)	0.0239 (10)	0.0002 (8)	0.0044 (9)
C3	0.0480 (12)	0.0438 (13)	0.0328 (11)	0.0263 (11)	−0.0014 (9)	0.0041 (9)
C4	0.0556 (14)	0.0524 (15)	0.0333 (11)	0.0272 (12)	0.0097 (10)	0.0090 (10)
C5	0.0422 (12)	0.0347 (12)	0.0508 (13)	0.0190 (10)	0.0108 (10)	0.0086 (10)
C6	0.0423 (12)	0.0411 (13)	0.0437 (13)	0.0163 (10)	−0.0014 (10)	−0.0049 (10)
C7	0.0663 (17)	0.0717 (19)	0.0387 (13)	0.0360 (15)	−0.0145 (12)	−0.0003 (12)
C8	0.0371 (11)	0.0488 (14)	0.0366 (11)	0.0238 (10)	−0.0010 (9)	0.0071 (9)
C9	0.0473 (13)	0.0781 (19)	0.0333 (12)	0.0367 (13)	0.0062 (10)	0.0024 (12)

Geometric parameters (Å, º)

O1—C5	1.211 (2)	O3—H301	0.948
O2—C8	1.426 (3)	C2—H21	0.970
O3—C9	1.395 (2)	C2—H22	0.970
C1—C2	1.530 (3)	C4—H4	0.930
C1—C6	1.512 (2)	C6—H61	0.970
C1—C8	1.534 (4)	C6—H62	0.970
C1—C9	1.541 (3)	C7—H71	0.960
C2—C3	1.480 (3)	C7—H72	0.960
C3—C4	1.351 (3)	C7—H73	0.960
C3—C7	1.495 (3)	C8—H81	0.970
C4—C5	1.445 (3)	C8—H82	0.970
C5—C6	1.531 (3)	C9—H91	0.970
O2—H201	0.915	C9—H92	0.970
C2—C1—C6	109.76 (18)	C3—C4—H4	118.6

C2—C1—C8	109.12 (18)	C5—C4—H4	118.6
C2—C1—C9	109.4 (2)	C1—C6—H61	108.4
C6—C1—C8	110.8 (2)	C1—C6—H62	108.4
C6—C1—C9	108.79 (18)	C5—C6—H61	108.4
C8—C1—C9	109.0 (2)	C5—C6—H62	108.4
C1—C2—C3	115.5 (2)	H61—C6—H62	109.5
C2—C3—C4	121.45 (19)	C3—C7—H71	109.5
C2—C3—C7	115.92 (19)	C3—C7—H72	109.5
C4—C3—C7	122.6 (2)	C3—C7—H73	109.5
C3—C4—C5	122.9 (2)	H71—C7—H72	109.5
O1—C5—C4	122.5 (2)	H71—C7—H73	109.5
O1—C5—C6	120.8 (2)	H72—C7—H73	109.5
C4—C5—C6	116.7 (2)	O2—C8—H81	108.6
C1—C6—C5	113.84 (17)	O2—C8—H82	108.6
O2—C8—C1	113.12 (18)	C1—C8—H81	108.6
O3—C9—C1	113.6 (2)	C1—C8—H82	108.6
C8—O2—H201	105.8	H81—C8—H82	109.5
C9—O3—H301	103.4	O3—C9—H91	108.4
C1—C2—H21	107.9	O3—C9—H92	108.4
C1—C2—H22	107.9	C1—C9—H91	108.4
C3—C2—H21	107.9	C1—C9—H92	108.4
C3—C2—H22	107.9	H91—C9—H92	109.5
H21—C2—H22	109.5		

C2—C1—C6—C5	−50.0 (3)	C8—C1—C9—O3	−58.2 (2)
C6—C1—C2—C3	44.7 (3)	C9—C1—C8—O2	−59.9 (2)
C2—C1—C8—O2	−179.24 (16)	C1—C2—C3—C4	−19.9 (4)
C8—C1—C2—C3	−76.9 (2)	C1—C2—C3—C7	161.0 (2)
C2—C1—C9—O3	61.0 (3)	C2—C3—C4—C5	−1.2 (4)
C9—C1—C2—C3	164.0 (2)	C7—C3—C4—C5	177.8 (3)
C6—C1—C8—O2	59.8 (2)	C3—C4—C5—O1	176.3 (3)
C8—C1—C6—C5	70.6 (2)	C3—C4—C5—C6	−4.6 (4)
C6—C1—C9—O3	−179.1 (2)	O1—C5—C6—C1	−149.4 (2)
C9—C1—C6—C5	−169.7 (2)	C4—C5—C6—C1	31.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H201···O3^{i}	0.92	1.85	2.738 (2)	163
O3—H301···O2^{ii}	0.95	1.85	2.733 (2)	155

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