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1,1′,1″-[(5*RS*,6*RS*)-6-Hydroxy-6-methyl-3,4,5,6-tetrahydro-2*H*-pyran-3,3,5-triyl]triethanone

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In the title compound, $C_{12}H_{18}O_5$, the ring has a chair conformation, with endocyclic torsion angle magnitudes in the range 47.7 (3)–66.7 (2)°. The OH group donates an intermolecular hydrogen bond to a C═O group with an O···O distance of 2.791 (3) Å, forming chains.

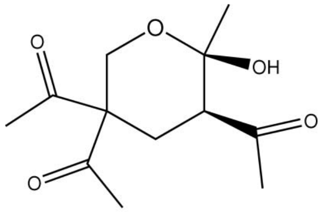
Key indicators

Single-crystal X-ray study
T = 120 K
Mean *σ*(C–C) = 0.004 Å
R factor = 0.046
wR factor = 0.106
Data-to-parameter ratio = 11.3

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

Comment

Since the early work of Knoevenagel (1894) in the late nineteenth century, the condensation products of 2,4-pentanedione with aldehydes have been investigated. Kennedy & McMurry (1969) determined that when acetylacetone is condensed according to the Scholtz (1897) procedure, using a 1:1 molar ratio, the title compound is exclusively formed. In our efforts to construct linkers and nodes as precursors for the preparation of molecular solids, the title compound, (I), was obtained.



(I)

The endocyclic torsion angles (Table 1) indicate a chair conformation for the ring. Two of the MeCO groups, C9 and C11, have their carbonyls essentially eclipsed with ring CH_2 groups, while the third, C7, has its carbonyl nearly eclipsed with a ring C—H group. That carbonyl, C7═O3, accepts an intermolecular hydrogen bond. The O—H···O hydrogen bond (Table 2) is nearly linear, and forms chains in the [101] direction.

Experimental

The title compound was synthesized according to the procedure of Aarna *et al.* (1975), using an excess of formaldehyde. A solution of the product in diethyl ether was cooled in a dry ice–acetone bath, producing crystals suitable for X-ray analysis.

Crystal data

$C_{12}H_{18}O_5$
M_r = 242.26
Monoclinic, *Pn*
a = 7.598 (2) Å
b = 8.813 (3) Å
c = 9.169 (2) Å
β = 91.26 (2)°
V = 613.8 (3) Å^3
Z = 2
Mo *Kα* radiation
μ = 0.10 mm^{-1}
T = 120 K
0.46 × 0.07 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer with an Oxford Cryosystems Cryostream cooler
Absorption correction: none
6461 measured reflections
1796 independent reflections
1588 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.106$
$S = 1.07$
1796 reflections
159 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{\rm max} = 0.45$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C5	1.424 (3)	O2—C1	1.410 (3)
O1—C1	1.432 (3)		
C5—O1—C1	113.62 (18)		
C5—O1—C1—C2	60.4 (2)	C3—C4—C5—O1	59.2 (2)
O1—C1—C2—C3	−50.0 (2)	C1—C2—C7—O3	−92.7 (3)
C1—C2—C3—C4	47.7 (3)	C3—C4—C9—O4	9.1 (3)
C2—C3—C4—C5	−51.1 (3)	C5—C4—C11—O5	7.7 (3)
C1—O1—C5—C4	−66.7 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O3$^{\rm i}$	0.84	1.96	2.791 (3)	170

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

H atoms on C were placed in idealized positions, with C—H distances in the range 0.98–1.00 Å and thereafter treated as riding. $U_{\rm iso}$ values for H atoms were set at 1.2 times $U_{\rm eq}$ of the attached C atoms (1.5 for methyl). A torsion parameter was refined for each methyl group and the OH group. In the absence of significant anomalous scattering, Friedel pairs were averaged.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK*; data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

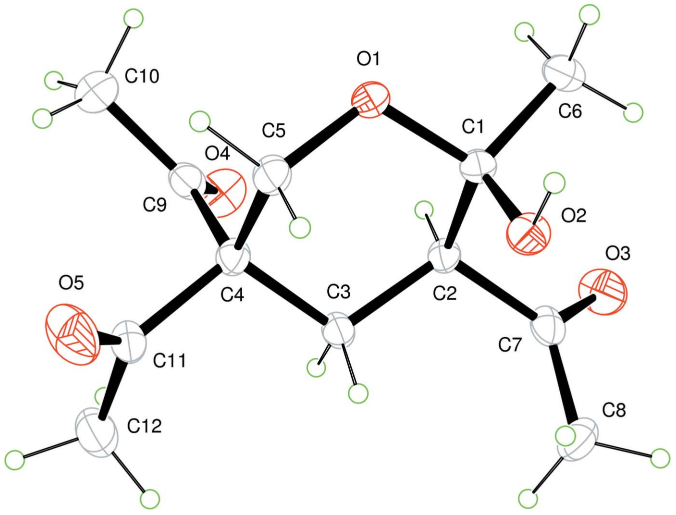


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level. H atoms are shown with arbitrary radius.

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