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Methyl 2-hydroxy-2-[5′-hydroxy-5′-(1-hydroxy-1-methylethyl)-5,2′-dimethylperhydro-2,2′-bifuranyl-5-yl]acetate

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Methyl 2-hydroxy-2-[5′-hydroxy-5′-(1-hydroxy-1-methylethyl)-5,2′-dimethyl-perhydro-2,2′-bifuranyl-5-yl]acetate

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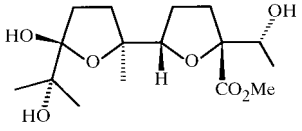
The relative configuration was determined for the title compound, $C_{16}H_{28}O_7$, which was obtained as a mixture of epimers. There are both intra- and intermolecular hydrogen bonds, the latter forming dimers around the crystallographic centres of inversion.

Comment

The title molecule, (I), is involved in both intra- and intermolecular hydrogen bonding. The intramolecular hydrogen bonds are O3—H03···O6 and O7—H07···O5. Both have donor–acceptor distances of less than 2.73 Å, but the O3—H03···O6 angle of 166 (2)° suggests it is the stronger.



(I)

The intermolecular hydrogen bonds are bifurcated at H06, *i.e.* O6—H06···O2 and O6—H06···O3. These link pairs of molecules to from dimers situated around the crystallographic centres of inversion, and the action of these centres on the chiral molecule means they are enantiomerically related.

Experimental

Oxidation of (*E,E*)-methyl farnesoate by potassium permanganate afforded a mixture of epimeric lactols in a ratio of 6:1. Crystallization from an ether–hexane mixture afforded X-ray quality crystals of the racemate, confirming the proposed stereochemical outcome of the oxidative cyclization reaction. We cannot tell if (I) (m.p. 353–356 K) is the major epimer as the two epimers may interconvert *via* the ring-open form in solution. Crystallization from an ether–hexane mixture afforded X-ray quality crystals of the racemate, confirming the proposed stereochemical outcome of the oxidative cyclization reaction (Brown *et al.*, 2000).

Crystal data

$C_{16}H_{28}O_7$	D_x = 1.264 Mg m^{-3}
M_r = 332.38	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 11814 reflections
a = 7.5615 (2) Å	θ = 3.24–25.03°
b = 11.1023 (4) Å	μ = 0.098 mm^{-1}
c = 20.9017 (5) Å	T = 150 (2) K
β = 95.302 (2)°	Block, colourless
V = 1747.19 (9) Å^3	0.4 × 0.4 × 0.3 mm
Z = 4	

Data collection

Nonius KappaCCD area-detector diffractometer	2392 reflections with $I > 2\sigma(I)$
Φ and ω scans to fill Ewald sphere	R_{int} = 0.026
Absorption correction: empirical (*SORTAV*; Blessing, 1997)	θ_{max} = 25.03°
T_{min} = 0.962, T_{max} = 0.971	$h = -9 \rightarrow 8$
5490 measured reflections	$k = -13 \rightarrow 13$
2876 independent reflections	$l = -23 \rightarrow 24$
	Intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 0.5111P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R(F)$ = 0.038	$(\Delta/\sigma)_{max}$ = 0.002
$wR(F^2)$ = 0.100	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
S = 1.04	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
2876 reflections	Extinction correction: *SHELXL*97
321 parameters	Extinction coefficient: 0.016 (3)
All H-atom parameters refined	

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H03···O6	0.88 (2)	1.86 (3)	2.7250 (16)	166 (2)
O6—H06···O2^{i}	0.87 (3)	2.08 (3)	2.9381 (16)	171 (2)
O6—H06···O3^{i}	0.87 (3)	2.30 (3)	2.7973 (16)	116 (2)
O7—H07···O5	0.88 (3)	2.24 (3)	2.7048 (17)	113 (2)

Symmetry code: (i) $1-x, 2-y, -z$.

The completeness for θ_{max} = 25.03° was 93.6%, which was slightly lower than the requirement (>95%). The H-atom positions were located from the difference map and fully refined. The bond-length ranges were C—H 0.94 (3)–1.04 (3) Å and O—H 0.87 (3)–0.88 (2) Å.

Data collection and cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); data reduction: *DENZO*, *COLLECT* and *maXus* (Mackay *et al.*, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *WINGX* (Farrugia, 1998).

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