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Ethyl (2*S**)-2-[(2*R**,2′*R**,5*S**)-2′,5-dimethyl-5′-oxoperhydro-[2,2′]]bifuranyl-5-yl]-2-hydroxyethanoate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.030
wR factor = 0.075
Data-to-parameter ratio = 9.2

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

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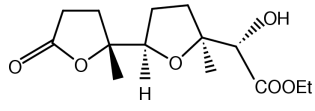
The molecular structure of the title compound, $C_{14}H_{22}O_6$, has four chiral centres, for which only the relative configuration has been unequivocally determined. The molecules form a supramolecular array of infinite one-dimensional chains.

Comment

The title compound, (I) (Fig. 1), was synthesized as part of a study of the $KMnO_4$-mediated oxidative cyclization of 1,5,9-trienes (Brown *et al.*, 2002). The molecule is composed of two substituted furan moieties connected to each other at the 2- and 5-positions, and exhibits bond lengths and angles consistent with expected values (Orpen *et al.*, 1992) derived from structures in the Cambridge Structural Database (Allen, 2002).



(I)

The molecular structure of (I) contains two furan rings which, from puckering analysis (Cremer & Pople, 1975), adopt envelope (about C3) and twisted (about C7—C8) conformations. The molecule contains four chiral centres which, for the given absolute configuration, are C4 = *R*, C6 = *R*, C9 = *S* and C11 = *S*.

The crystal structure is a one-dimensional chain arising from a hydrogen-bonded O4—H4···O1^{i} interaction [symmetry code: (i) $-x$, $-y$, $z+\frac{1}{2}$], with a donor–acceptor separation of 2.8931 (16)Å.

Experimental

Ethyl (2*Z*,6*E*)-3,7,11-trimethyl-2,6,10-dodecatrienoate was oxidized with $KMnO_4$ followed by $Pb(OAc)_4$ to afford the title compound, (I), as a colourless oil which solidified on standing (Brown *et al.*, 2002). Recrystallization from ethyl acetate/hexane gave colourless plates suitable for X-ray structure determination.

Crystal data

$C_{14}H_{22}O_6$
M_r = 286.32
Orthorhombic, $Pna2_1$
a = 9.3133 (3) Å
b = 15.4441 (4) Å
c = 9.8424 (3) Å
V = 1415.69 (7) Å^3
Z = 4
D_x = 1.343 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 15124 reflections
θ = 2.9–27.5°
μ = 0.10 mm^{-1}
T = 120 (2) K
Plate, colourless
0.26 × 0.22 × 0.10 mm

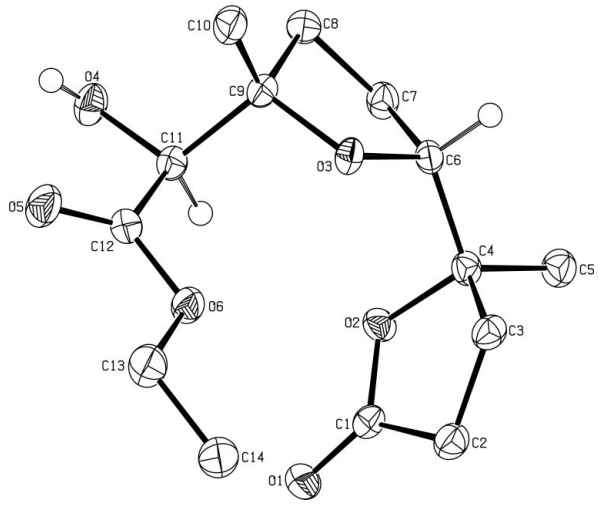


Figure 1
View of (I) (50% probability displacement ellipsoids), with specific H atoms retained to show the relative configuration.

Data collection

Bruker–Nonius KappaCCD diffractometer	1717 independent reflections
φ and ω scans	1555 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)	$R_{int} = 0.061$
$T_{min} = 0.973$, $T_{max} = 0.990$	$\theta_{max} = 27.5^\circ$
14735 measured reflections	$h = -10 \rightarrow 12$
	$k = -19 \rightarrow 20$
	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0417P)^2 + 0.1548P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.030$	$(\Delta/\sigma)_{max} = 0.005$
$wR(F^2) = 0.075$	$\Delta\rho_{max} = 0.17$ e Å^{-3}
$S = 1.08$	$\Delta\rho_{min} = -0.17$ e Å^{-3}
1717 reflections	Extinction correction: *SHELXL*97
186 parameters	Extinction coefficient: 0.009 (2)
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O1^{i}	0.84	2.10	2.8931 (16)	157

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

Compound (I) crystallized in the non-centrosymmetric space group $Pna2_1$; however, due to the insignificant anomalous scattering, the Flack (1983) parameter refined is indeterminate and so Friedel pairs were merged before the final refinement. H atoms are included in constrained positions, with torsion angles allowed to freely refine in the case of methyl and hydroxy groups.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003).

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