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(1*R*,2*S*,3*R*,6*S*,7*R*,8*S*)-Tricyclo[6.2.1.$0^{2,7}$]-undeca-4,9-diene-3,6-diol

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The asymmetric unit of the title compound, $C_{11}H_{14}O_2$, contains two molecules. The alcohol groups have *cis* stereochemistry and are involved in hydrogen bonding (acting as both donors and acceptors), resulting in the formation of an infinite three-dimensional network.

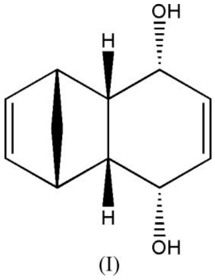
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.052
wR factor = 0.118
Data-to-parameter ratio = 14.3

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

Comment

We are currently investigating trishomocubane derivatives with a high affinity for the sigma receptor (Liu *et al.*, 1999; 2001). The title compound, (I), was prepared as an intermediate in the synthesis of compounds intended to further explore the series. A crystal structure was obtained to confirm the relative stereochemistry of each alcohol group (C1, C4, C12, C15) after Luche reduction of the corresponding diketone. As expected, the reduction proceeds stereoselectively resulting in a *cis*-diol product (Fig. 1).



(I)

The precursor, (1*R*,2*S*,7*R*,8*S*)-tricyclo[6.2.1.$0^{2,7}$]undeca-4,9-diene-3,6-dione, is formed by a Diels–Alder reaction between 1,4-benzoquinone and cyclopentadiene, yielding the *endo* adduct (Cookson *et al.*, 1964; Yates & Switlak, 1990), the stereochemistry of which is not affected by the subsequent reduction of the ketone groups.

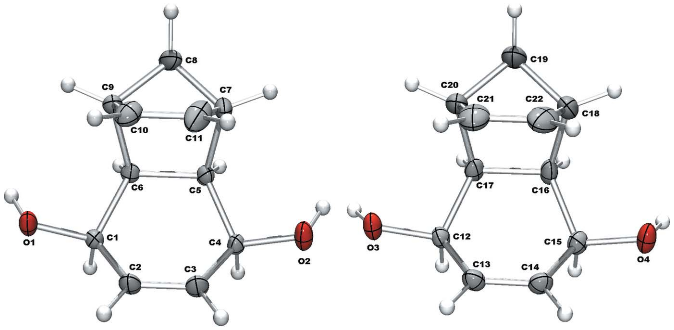


Figure 1
The structures of the two crystallographically independent but chemically identical molecules in the asymmetric unit, shown with 50% probability ellipsoids.

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The alcohol groups are involved in hydrogen bonding with each O atom acting as both a donor and an acceptor to form an infinite three-dimensional network (Fig. 2 and Table 1). If each alcohol is defined as a node and the rest of the molecule as a linear connector, Wellsian analysis (Wells, 1977) suggests that the topology is a non-uniform (10,3)-type network. A depiction of the shortest loop is shown in Fig. 2. Owing to the distortion of the network it was not possible to assign the topology to one of the *a*–*g* subgroups of the (10,3) group and this network may represent a new topology.

Experimental

(1*R*,2*S*,7*R*,8*S*)-Tricyclo[6.2.1.0^{2,7}]undeca-4,9-diene-3,6-dione was prepared by a published procedure (Marchand & Allen, 1974). A Luche reduction of this compound was performed according to the literature procedure (Marchand *et al.*, 1986). Slow evaporation of an acetone solution of (I) afforded the large colourless prismatic crystals used for this study.

Crystal data

$C_{11}H_{14}O_2$
$M_r = 178.22$
Orthorhombic, $Pca2_1$
$a = 12.5265$ (15) Å
$b = 10.7003$ (13) Å
$c = 13.9454$ (13) Å
$V = 1869.2$ (4) Å^3

$Z = 8$
$D_x = 1.267$ Mg m$^{-3$}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 150$ (2) K
Prism, colourless
0.30 × 0.28 × 0.12 mm

Data collection

Bruker–Nonius APEXII diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{min} = 0.766$, $T_{max} = 0.990$

21673 measured reflections
3539 independent reflections
2747 reflections with $I > 2\sigma(I)$
$R_{int} = 0.063$
$\theta_{max} = 32.7°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.118$
$S = 1.03$
3539 reflections
247 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0599P)^2 + 0.0703P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O*···O2^{i}	0.87 (3)	1.86 (3)	2.730 (2)	175 (3)
O3—H3*O*···O1^{ii}	0.90 (3)	1.82 (3)	2.720 (2)	171 (3)
O4—H4*O*···O3^{iii}	0.80 (3)	1.93 (3)	2.731 (2)	175 (3)
O2—H2*O*···O4	0.81 (3)	1.97 (3)	2.777 (2)	174 (3)

Symmetry codes: (i) $x+\frac{1}{2}, -y+1, z$; (ii) $-x+\frac{3}{2}, y-1, z+\frac{1}{2}$; (iii) $x-\frac{1}{2}, -y, z$.

Carbon-bound H atoms were included in idealized positions and refined using a riding model. Methine, aromatic and methylene C—H bond lengths were fixed at 1.00, 0.95 and 0.99 Å, respectively. U_{iso}(H) values were fixed at 1.2U_{eq} of the parent C atoms. Alcohol H atoms were located in a difference Fourier map and refined with U_{iso}(H) values fixed at 0.038 Å^2. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

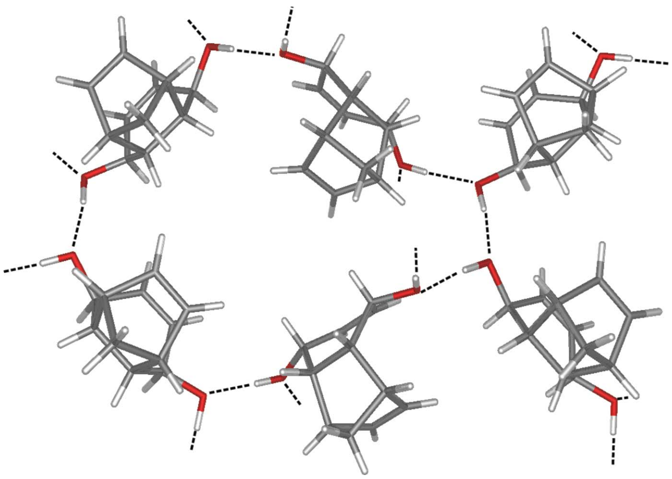

Figure 2
A section of the three-dimensional hydrogen-bonding network. Hydrogen bonds are shown as dashed lines

Data collection: *APEX2* (Bruker–Nonius, 2003); cell refinement: *SAINT* (Bruker–Nonius, 2003); data reduction: *SAINT* and *XPREP* (Bruker–Nonius, 2003); 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), *WinGX32* (Farrugia, 1999), *POV-RAY 3.5* (Cason, 2002) and *WebLab ViewerPro 3.7* (Molecular Simulations, 2000); software used to prepare material for publication: *enCIFer* (Version 1.1; Allen *et al.*, 2004).

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