

(1*S*,3*R*,4*R*,4*aS*,8*aS*)-4-[(3*S*)-3-Hydroxy-3-methyl-4-pentenyl]-3,4*a*,8,8-tetramethyldecahydro-1,3-naphthalenediol

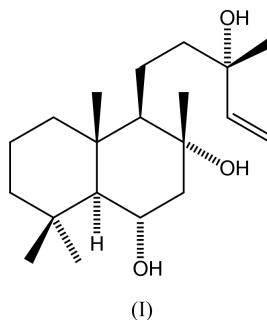
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h.kooijman@chem.uu.nl**Key indicators**Single-crystal X-ray study
 $T = 150\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.055
 wR factor = 0.107
Data-to-parameter ratio = 11.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{20}\text{H}_{36}\text{O}_3$, forms clusters of molecules connected by a co-operative hydrogen-bond chain of five members. The direction of the chain is disordered due to the presence of a crystallographic twofold rotation axis. The clusters are linked *via* hydrogen bonds in an infinite two-dimensional network.

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The structure determination of the title compound, (I), was carried out to confirm the absolute configuration of atom C8, relative to the known configuration of the other chiral centres (Bolster, Jansen & de Groot, 2001; Bolster, Lagnel *et al.*, 2001).



The crystal packing displays clusters of four molecules, connected by hydrogen bonds (see Fig. 2). Owing to the arrangement of acceptor/donor atoms in a linear chain, it is not possible for each donor to be involved in a hydrogen bond; some acceptor atoms will also remain free. Since a co-operative hydrogen-bond chain is normally preferred for this arrangement of donor/acceptor atoms, the H atoms of the hydroxyl groups were placed in positions enabling such a pattern. The central $\text{O}3 \cdots \text{O}3$ hydrogen bond is located on a crystallographic twofold rotation axis, causing the H-atom positions to be disordered, as is illustrated in Fig. 2. The direction of the hydrogen-bonded chain is reversed by the rotation axis. Since each of the hydrogen bonds is chemically different from all others, the primary graph set of the co-operative chain is *DDDDD* (Bernstein *et al.*, 1995).

Hydrogen bonds also link molecules which are related by crystallographic 2_1 screw axes into infinite chains [graph set $C(6)$, see Fig. 2], running parallel to the *b* axis. The disordered hydrogen bonds forming these infinite chains are also part of the five-membered cooperative chain.

The combination of finite co-operative hydrogen-bonded chains and infinite chains related to the screw axes creates an infinite two-dimensional network, with base vectors *a* and *b*.

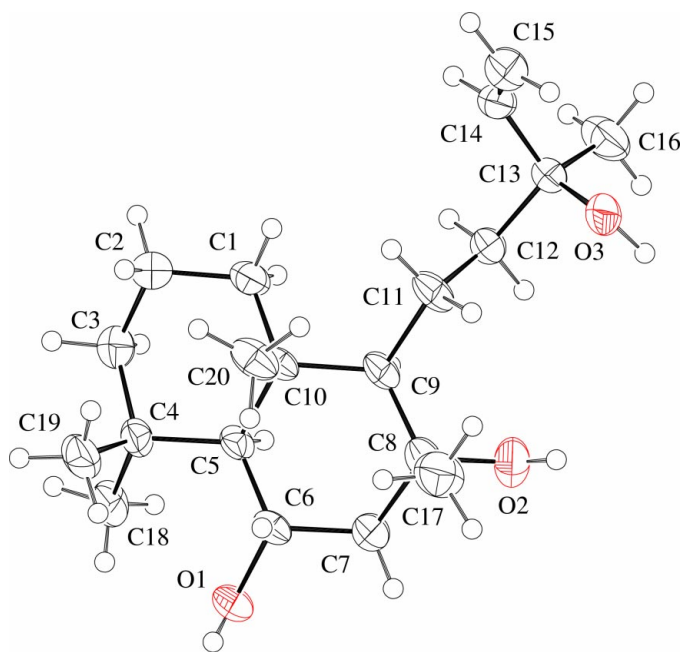


Figure 1
Displacement ellipsoid plot of the title compound drawn at the 50% probability level (Spek, 2001).

Experimental

Crystals were obtained from a solution in ethyl acetate and CH_2Cl_2 . Full details of synthesis, crystallization and characterization with IR, NMR and HRMS are given by Bolster, Jansen & de Groot (2001).

Crystal data

$\text{C}_{20}\text{H}_{36}\text{O}_3$	$D_x = 1.140 \text{ Mg m}^{-3}$
$M_r = 324.49$	Mo $K\alpha$ radiation
Monoclinic, C2	Cell parameters from 293 reflections
$a = 24.767 (3) \text{ \AA}$	$\theta = 2.0\text{--}25.0^\circ$
$b = 6.2051 (10) \text{ \AA}$	$\mu = 0.07 \text{ mm}^{-1}$
$c = 14.1446 (15) \text{ \AA}$	$T = 150 \text{ K}$
$\beta = 119.55 (5)^\circ$	Block, colorless
$V = 1891.0 (10) \text{ \AA}^3$	$0.30 \times 0.30 \times 0.20 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD area-detector diffractometer	$R_{\text{int}} = 0.042$
φ scans, and ω scans with κ offsets	$\theta_{\text{max}} = 27.5^\circ$
28 559 measured reflections	$h = -32 \rightarrow 32$
2367 independent reflections	$k = -8 \rightarrow 8$
2271 reflections with $I > 2\sigma(I)$	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.01P)^2 + 3P]$
$R[F^2 > 2\sigma(F^2)] = 0.055$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.107$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.40 \text{ e \AA}^{-3}$
2367 reflections	$\Delta\rho_{\text{min}} = -0.21 \text{ e \AA}^{-3}$
213 parameters	
H atoms: see text	

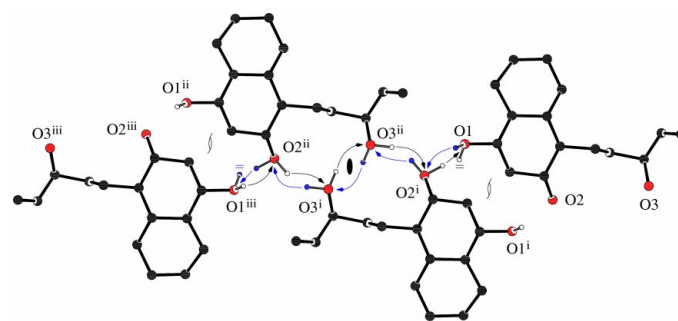


Figure 2
The hydrogen bonding in the title compound. H atoms not involved in the discussion, and the methyl moieties C16 through C20, have been omitted for clarity. Hydrogen bonds are indicated with arrows. Black arrows and white H atoms are used for one of the disorder components; blue arrows and blue H atoms for the other (symmetry-related) component. The positions of rotation and screw axes are indicated. The termination of the co-operative chain is indicated with =. [Symmetry codes: (i) $3/2-x, 1/2+y, 2-z$; (ii) $-1/2+x, 1/2+y, z$; (iii) $1-x, y, 2-z$.]

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1—C6	1.435 (4)	O3—C13	1.432 (4)
O2—C8	1.493 (5)		
O1—C6—C5	112.3 (3)	O2—C8—C9	108.1 (3)
O1—C6—C7	107.7 (3)	O3—C13—C16	110.2 (3)
O2—C8—C17	105.0 (3)	O3—C13—C14	109.7 (3)
O2—C8—C7	105.9 (3)	O3—C13—C12	108.3 (2)

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O1—H11 \cdots O2 ⁱ	0.96	2.01	2.817 (4)	140
O2—H21 \cdots O3 ⁱⁱ	0.96	1.95	2.728 (4)	136
O2—H22 \cdots O1 ⁱⁱⁱ	0.96	1.93	2.817 (4)	152
O3—H31 \cdots O3 ⁱⁱ	0.96	1.99	2.701 (3)	129
O3—H32 \cdots O2 ⁱⁱ	0.96	1.92	2.728 (4)	141
C11—H11B \cdots O3	0.99	2.53	2.912 (4)	103
C18—H18A \cdots O1	0.98	2.29	2.790 (5)	111

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

Owing to a lack of significant anomalous scatterers, the absolute configuration could not be determined *ab initio*. The chirality was chosen in agreement with the well known stereochemistry of atoms C9 (*R*) and C10 (*S*). 1078 Friedel pairs were measured; these Friedel opposites were merged before refinement. The hydroxyl H atoms were introduced at positions favourable for the formation of hydrogen bonds (see *Comment*). Their coordinates were included in rigid groups with the oxygen carrier atom as pivot atom. The idealized H atoms of each methyl moiety were included in rigid groups with the carbon carrier atom and were allowed to rotate around the C—C bond. All other H atoms were included at calculated positions, riding on their carrier atoms. Isotropic displacement parameters of the H atoms were coupled to the equivalent isotropic displacement parameter of their carrier atoms by a fixed factor.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*;

program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985);
program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997);
molecular graphics: *PLATON* (Spek, 2001); software used to prepare
material for publication: *PLATON*.

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