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86.7 (3)° with the mean plane through all the C atoms of both rings. The planarity is in part facilitated by an intramolecular hydrogen bond: O2$\cdots$H1 distance 2.102 Å and O1—H1$\cdots$O2 angle 117.0°. Close intermolecular contacts, which range from 2.412 to 2.780 Å, exist between both O atoms and various alkyl protons.

(Z)-12-Hydroxy-14,15-dinorlabda-8(17),11-dien-13-one, C18H28O2

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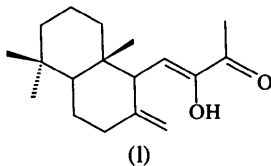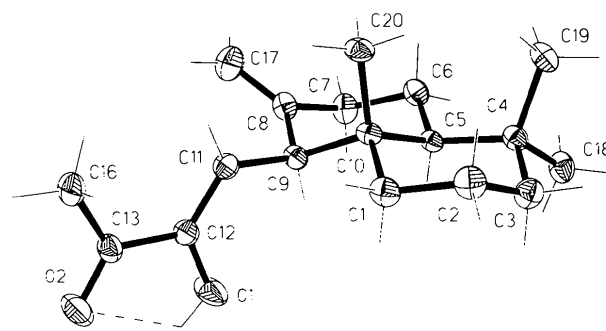*Department of Chemistry, University of Otago, PO Box 56, Dunedin, New Zealand*

Fig. 1. Molecular structure of (1); displacement ellipsoids are drawn with boundary surfaces at the 50% level (Johnson, 1965).

Abstract

The stereochemistry of the diosphenol moiety of the title compound [alternative systematic name: 4-(decahydro-5,5,8a-trimethyl-2-methylene-1-naphthyl)-3-hydroxy-3-buten-2-one] was confirmed to have the Z configuration. The diosphenol moiety is approximately planar with an intramolecular hydrogen bond existing between the alcohol and the keto group.

Comment

As part of investigations to prepare potential odorants from labda-8(17),14-dien-13-ol (Grant, Hanton, Lynch, Robinson & Wong, 1994), we isolated a series of stereochemically related diosphenols (Wong, 1990). The crystal structure determination of (Z)-12-hydroxy-14,15-dinorlabda-8(17),11-dien-13-one, (1), determined the stereochemistry of these diosphenols to be the Z configuration.

(1)

In (1), the six-membered rings adopt flattened chair conformations. Me groups C19 and C20 adopt 1,3-diaxial positions and are splayed outwards from the rings by 13 (1)°. The diosphenol moiety C9,C11,C12,C13,C16,O1,O2 is approximately planar [average deviation 0.052 (6) Å] and makes an angle of

Experimental

14,15-Dinorlabd-8(17)-en-13-one (2.5 g) in 1*M* ^{t}BuOK/^{t}BuOH (100 ml) was shaken with oxygen until the uptake was complete. Acidification with 2*M* HCl followed by standard workup procedures and radial chromatography (5% ether/hexane) gave (1) (1.17 g, 44%) (Wong, 1990). Crystals were obtained by slow evaporation from hexane.

Crystal data

C18H28O2	Mo $K\alpha$ radiation
$M_r = 276.4$	$\lambda = 0.71069$ Å
Monoclinic	Cell parameters from 25
$P2_1$	reflections
$a = 7.353\,(9)$ Å	$\theta = 12–31°$
$b = 10.805\,(7)$ Å	$\mu = 0.073$ mm^{-1}
$c = 9.959\,(11)$ Å	$T = 148\,(2)$ K
$\beta = 93.2\,(1)°$	Rectangular plate
$V = 790\,(2)$ Å^3	$0.82 \times 0.56 \times 0.20$ mm
$Z = 2$	Colourless
$D_x = 1.162$ Mg m^{-3}	

Data collection

Nicolet *R*3*m* diffractometer	$\theta_{max} = 32.49°$
ω scans	$h = -11 \rightarrow 11$
Absorption correction:	$k = 0 \rightarrow 16$
none	$l = 0 \rightarrow 15$
2983 measured reflections	3 standard reflections
2983 independent reflections	monitored every 97
2361 observed reflections	reflections
[$I > 2\sigma(I)$]	intensity decay: 5%

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.244$ e Å^{-3}
$R[F^2 > 2\sigma(F^2)] = 0.0517$	$\Delta\rho_{min} = -0.243$ e Å^{-3}
$wR(F^2) = 0.1357$	Extinction correction: none

$S = 1.020$
2983 reflections
184 parameters
H-atom parameters not
    refined
$w = 1/[\sigma^2(F_o^2) + (0.1000P)^2]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.020$

Atomic scattering factors
from *International Tables
for Crystallography* (1992,
Vol. C, Tables 4.2.6.8 and
6.1.1.4)
Absolute configuration:
inferred from preparation

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* ($\AA^2$)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.7576 (3)	0.6839 (3)	0.6587 (2)	0.0434 (5)
HO1	0.7910 (3)	0.7445 (3)	0.6171 (2)	
O2	0.9975 (3)	0.7165 (3)	0.4826 (2)	0.0374 (5)
C1	0.9081 (3)	0.4926 (3)	0.9822 (2)	0.0245 (4)
C2	0.9611 (3)	0.4671 (3)	1.1304 (2)	0.0270 (4)
C3	0.7945 (3)	0.4788 (3)	1.2138 (2)	0.0266 (4)
C4	0.6363 (3)	0.3930 (3)	1.1689 (2)	0.0203 (4)
C5	0.5933 (2)	0.4082 (3)	1.0141 (2)	0.0170 (3)
C6	0.4375 (3)	0.3259 (3)	0.9567 (2)	0.0231 (4)
C7	0.3778 (3)	0.3621 (3)	0.8121 (2)	0.0264 (5)
C8	0.5385 (3)	0.3681 (3)	0.7247 (2)	0.0237 (4)
C9	0.6885 (3)	0.4527 (3)	0.7816 (2)	0.0197 (4)
C10	0.7600 (2)	0.4044	0.9241 (2)	0.0174 (3)
C11	0.8373 (3)	0.4749 (3)	0.6874 (2)	0.0213 (4)
C12	0.8646 (3)	0.5851 (3)	0.6315 (2)	0.0224 (4)
C13	1.0032 (3)	0.6136 (3)	0.5339 (2)	0.0231 (4)
C16	1.1478 (3)	0.5228 (3)	0.5024 (2)	0.0274 (5)
C17	0.5465 (4)	0.3016 (3)	0.6135 (3)	0.0354 (6)
C18	0.4705 (3)	0.4353 (3)	1.2435 (2)	0.0275 (5)
C19	0.6801 (3)	0.2596 (3)	1.2123 (2)	0.0268 (4)
C20	0.8414 (3)	0.2745 (3)	0.9087 (2)	0.0235 (4)

Table 2. *Selected geometric parameters* ($\AA$, °)

O1—C12	1.363 (3)	C6—C7	1.533 (3)
O2—C13	1.223 (3)	C7—C8	1.508 (3)
C1—C2	1.531 (3)	C8—C17	1.324 (3)
C1—C10	1.536 (3)	C8—C9	1.518 (3)
C2—C3	1.523 (3)	C9—C11	1.500 (3)
C3—C4	1.534 (3)	C9—C10	1.575 (3)
C4—C18	1.532 (3)	C10—C20	1.537 (3)
C4—C19	1.533 (3)	C11—C12	1.333 (3)
C4—C5	1.566 (3)	C12—C13	1.480 (3)
C5—C6	1.535 (3)	C13—C16	1.493 (3)
C5—C10	1.559 (3)		
C2—C1—C10	112.9 (2)	C11—C9—C8	113.7 (2)
C3—C2—C1	110.0 (2)	C11—C9—C10	113.5 (2)
C2—C3—C4	114.3 (2)	C8—C9—C10	109.6 (2)
C18—C4—C3	106.8 (2)	C20—C10—C1	109.4 (2)
C18—C4—C19	107.7 (2)	C20—C10—C5	113.9 (2)
C3—C4—C19	110.0 (2)	C1—C10—C5	109.3 (2)
C18—C4—C5	108.9 (2)	C20—C10—C9	109.0 (2)
C3—C4—C5	109.4 (2)	C1—C10—C9	109.1 (2)
C19—C4—C5	113.8 (2)	C5—C10—C9	105.96 (14)
C6—C5—C10	111.4 (2)	C12—C11—C9	122.4 (2)
C6—C5—C4	114.3 (2)	C11—C12—O1	120.9 (2)
C10—C5—C4	116.2 (2)	C11—C12—C13	125.8 (2)
C5—C6—C7	111.7 (2)	O1—C12—C13	113.2 (2)
C8—C7—C6	111.1 (2)	O2—C13—C12	117.1 (2)
C17—C8—C7	122.2 (2)	O2—C13—C16	121.2 (2)
C17—C8—C9	125.1 (2)	C12—C13—C16	121.7 (2)
C7—C8—C9	112.7 (2)		

The absolute stereochemistry of (1) could not be determined from the reflection data (Flack, 1983). The structure of the enantiomorph based on the stereochemistry of labda-8(17),14-dien-13-ol was refined and characterized. H atoms were placed in idealized positions and constrained to ride 0.96 $\AA$ from the appropriate C atom with fixed isotropic temperature factors.

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N,N-Dimethyl-1H-pyrrole-2-carboxamide

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Abstract

The low-temperature X-ray crystal structure of N,N-dimethyl-1H-pyrrole-2-carboxamide, $C_7H_{10}N_2O$, was determined. The molecular geometry indicates that the carbonyl π system interacts preferentially with the lone-pair electrons of the amide N atom rather than with the π system of the pyrrole ring. Intermolecular hydrogen bonds link the molecules into centrosymmetric dimers.