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### Isodrimenin, (5 $\alpha$ ,9 $\alpha$ )-4,5,5 $\alpha$ ,6,7,8,9,9 $\alpha$ -Octahydro-6,6,9 $\alpha$ -trimethylnaphthol[1,2-*c*]furan-1(3*H*)-one

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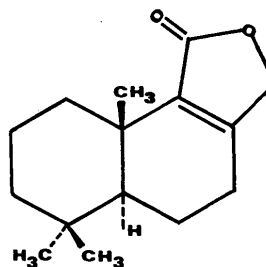
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**Abstract.** C<sub>15</sub>H<sub>22</sub>O<sub>2</sub>,  $M_r = 234.34$ , orthorhombic,  $P2_12_12_1$ ,  $a = 7.825$  (1),  $b = 23.669$  (2),  $c = 7.259$  (1) Å,  $V = 1344.5$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.10$  (1),  $D_x = 1.157$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.54178$  Å,  $\mu = 0.595$  mm<sup>-1</sup>,  $F(000) = 512$ ,  $T = 269$  K,  $R = 0.046$  for 1274 unique observed reflections with  $I > 2\sigma(I)$ . The molecule of this naturally occurring lactone exhibits a cyclohexane ring (chair) *trans* fused to a cyclohexene ring, with a five-membered lactone ring (planar) also fused to the cyclohexene ring. The structure consists of discrete molecules.

**Introduction.** The purpose in undertaking the structural analysis of natural isodrimenin (I) was to check the behavior of the two axial methyl groups on the cyclohexane ring, also present in drimenol. In the structure determination of the latter (Escobar & Wittke, 1984), the methyl H atoms were not resolved and the methyl C atoms exhibit a separation shorter than the normal van der Waals distance. Isodrimenin, like drimenol, was isolated from the bark of the Chilean canelo tree (*Drymis winteri* Forst). Its constitution and stereochemistry (I) were determined by chemical and spectroscopic methods (Appel, Connolly, Overton & Bond, 1960). Several total syntheses of this lactone and the isomeric drimenin, also naturally occurring, have been reported (Liapis, Ragoussis & Ragoussis, 1985)

and they have been used to prepare more biologically active members of the drimane class, such as warburganal.



**Experimental.** Colorless prismatic crystals,  $D_m$  measured with pycnometer and water; single crystal  $0.29 \times 0.53 \times 0.06$  mm used for X-ray analysis, Philips PW 1100 computer-controlled four-circle diffractometer, graphite monochromator, Cu  $K\alpha$  radiation,  $\omega/2\theta$  scan; cell parameters from least-squares refinement of setting angles of 66 strong reflections ( $11.9 \leq 2\theta \leq 85.5^\circ$ ); two standard reflections (022, 0 $\bar{2}\bar{2}$ ) every 76 measurements showed monotonic decay of intensity (total decay 2.6%), correction applied; 2770  $hkl$  and  $\bar{h}\bar{k}\bar{l}$  ( $1h|0/9$ ,  $1k|0/27$ ,  $1l|0/27$ ) up to  $(\sin\theta)/\lambda = 0.59$  Å<sup>-1</sup>, 1358 unique ( $R_{\text{int}} = 0.009$ ), 84

Table 1. Fractional atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$				
	<i>x</i>	<i>y</i>	<i>z</i>	$B_{eq}(\text{\AA}^2)$
O(1)	2733 (3)	2545 (1)	1694 (4)	6.0
O(2)	4682 (3)	2112 (1)	3436 (5)	7.6
C(1)	1678 (4)	1315 (1)	4775 (4)	3.9
C(2)	1704 (4)	1807 (1)	3456 (5)	4.0
C(3)	392 (4)	1999 (1)	2506 (5)	4.1
C(4)	-1380 (4)	1774 (2)	2610 (6)	5.1
C(5)	-1384 (5)	1211 (2)	3630 (6)	5.1
C(6)	-238 (4)	1244 (1)	5345 (5)	4.0
C(7)	-592 (5)	795 (1)	6859 (5)	5.0
C(8)	597 (6)	914 (2)	8484 (6)	6.2
C(9)	2480 (6)	979 (2)	7976 (6)	6.6
C(10)	2705 (5)	1444 (2)	6537 (6)	5.4
C(11)	3216 (4)	2145 (2)	2948 (6)	5.2
C(12)	930 (5)	2479 (2)	1317 (6)	5.3
C(13)	2488 (6)	814 (2)	3753 (6)	5.7
C(14)	-416 (7)	184 (2)	6207 (7)	7.1
C(15)	-2453 (6)	882 (2)	7568 (8)	7.4

unobserved with  $I \leq 2\sigma(I)$ ; Lp correction, absorption ignored.

Structure solved with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) with stereochemistry (I) for molecular scattering factor and *K* curve for scaling; full-matrix least-squares refinement on *F* with  $w = w_1 w_2$  ( $w_1 = 1$  if  $\sin\theta > 0.64$  otherwise  $w_1 = \sin\theta/0.64$ , and  $w_2 = 1$  if  $|F_o| < 10$  otherwise  $w_2 = 10/|F_o|$ ); all H atoms refined with isotropic (equivalent of the parent C atoms) temperature factor; 220 parameters varied, overdetermination ratio 5.8,  $S = 0.95$ ;  $R = 0.046$  and  $wR = 0.039$  for observed,  $R = 0.049$  for unique reflections; mean shift =  $0.03\sigma$ , max. shift =  $0.26\sigma$ ; final difference Fourier map excursions within  $-0.11$  and  $0.12 \text{ e \AA}^{-3}$ . Scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965) for H; computations with *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) and *NRC22* (Ahmed, Hall, Pippy & Huber, 1973) on an IBM 3031 computer at the Faculty Computer Centre.

**Discussion.** The labeling of the atoms is given in Fig. 1, the refined parameters in Table 1, and bond distances and angles in Table 2.\* The five-membered lactone ring is planar with maximum deviation of  $0.007$  ( $4$ )  $\text{\AA}$  and endocyclic torsion angles of  $-1.1$  ( $4$ ) to  $+1.0$  ( $4$ ) $^\circ$ . The cyclohexene ring has atoms C(1), C(2), C(3) and C(4) coplanar as expected [maximum deviation

\*Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond distances and bond angles concerning H atoms, mean planes and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44350 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Interatomic distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

C(1)–C(2)	1.509 (4)	C(6)–C(7)	1.555 (5)
C(1)–C(6)	1.563 (4)	C(7)–C(8)	1.528 (6)
C(1)–C(10)	1.541 (5)	C(7)–C(14)	1.529 (5)
C(1)–C(13)	1.535 (5)	C(7)–C(15)	1.558 (6)
C(2)–C(3)	1.318 (4)	C(8)–C(9)	1.526 (7)
C(2)–C(11)	1.473 (5)	C(9)–C(10)	1.527 (6)
C(3)–C(4)	1.487 (4)	C(11)–O(1)	1.368 (5)
C(3)–C(12)	1.488 (5)	C(11)–O(2)	1.203 (4)
C(4)–C(5)	1.525 (5)	C(12)–O(1)	1.446 (4)
C(5)–C(6)	1.536 (5)		
C(2)–C(1)–C(6)	105.3 (2)	C(1)–C(6)–C(7)	115.5 (3)
C(2)–C(1)–C(10)	111.5 (3)	C(5)–C(6)–C(7)	115.7 (3)
C(2)–C(1)–C(13)	106.5 (3)	C(6)–C(7)–C(8)	108.1 (3)
C(6)–C(1)–C(10)	107.6 (3)	C(6)–C(7)–C(14)	114.3 (3)
C(6)–C(1)–C(13)	116.2 (3)	C(6)–C(7)–C(15)	108.0 (3)
C(10)–C(1)–C(13)	109.8 (3)	C(8)–C(7)–C(14)	111.0 (3)
C(1)–C(2)–C(3)	126.0 (3)	C(8)–C(7)–C(15)	106.9 (3)
C(1)–C(2)–C(11)	126.0 (3)	C(14)–C(7)–C(15)	108.2 (3)
C(3)–C(2)–C(11)	107.9 (3)	C(7)–C(8)–C(9)	114.9 (3)
C(2)–C(3)–C(4)	125.2 (3)	C(8)–C(9)–C(10)	110.5 (4)
C(2)–C(3)–C(12)	110.3 (3)	O(1)–C(11)–O(2)	120.2 (3)
C(4)–C(3)–C(12)	124.5 (3)	O(1)–C(11)–C(2)	108.7 (3)
C(3)–C(4)–C(5)	109.8 (3)	O(2)–C(11)–C(2)	131.1 (3)
C(4)–C(5)–C(6)	110.4 (3)	O(1)–C(12)–C(3)	104.4 (3)
C(5)–C(6)–C(7)	115.7 (3)	C(11)–O(1)–C(12)	108.7 (3)
C(1)–C(6)–C(5)	110.5 (3)	C(9)–C(10)–C(1)	111.4 (3)

Possible hydrogen bonds ( $\text{\AA}, ^\circ$ )

<i>D</i> – <i>H</i> ... <i>A</i>	<i>D</i> – <i>H</i>	<i>H</i> ... <i>A</i>	<i>D</i> ... <i>A</i>	$\angle D$ – <i>H</i> ... <i>A</i>
C(4 <sup>iii</sup> )–H(41 <sup>iii</sup> )...O(2)	0.99 (3)	2.54 (3)	3.239 (4)	127 (2)
C(12 <sup>ii</sup> )–H(121 <sup>ii</sup> )...O(1)	1.12 (4)	2.58 (3)	3.322 (5)	122 (2)
C(8 <sup>vii</sup> )–H(81 <sup>vii</sup> )...C(3)	1.08 (3)	2.95 (3)	3.892 (5)	145 (2)
C(12 <sup>viii</sup> )–H(122 <sup>viii</sup> )...C(10)	1.00 (3)	3.00 (3)	3.911 (5)	152 (2)

Short intermolecular distances ( $\text{\AA}$ )

C(12)...C(4 <sup>ii</sup> )	3.960 (6)	C(14 <sup>iii</sup> )...H(151 <sup>v</sup> )	3.08 (4)
C(11)...C(12 <sup>ii</sup> )	3.858 (6)		
C(13 <sup>iii</sup> )...C(14 <sup>iv</sup> )	3.774 (7)		

Symmetry code: (ii)  $\frac{1}{2} + x, \frac{1}{2} - y, -z$ ; (iii)  $1 + x, y, z$ ; (iv)  $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (v)  $-x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (vi)  $\frac{1}{2} - x, -y, \frac{1}{2} + z$ ; (vii)  $x, y, -1 + z$ ; (viii)  $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$ .

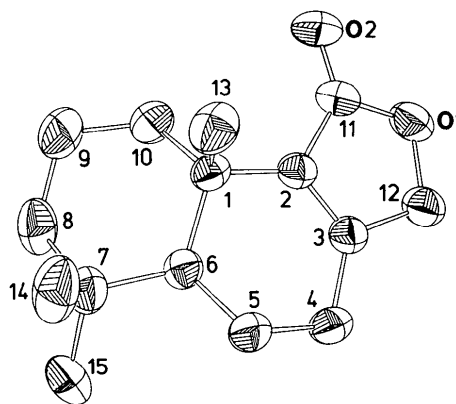


Fig. 1. The isodrimenin molecule with H atoms omitted and 40% probability thermal ellipsoids.

$0.008$  ( $3$ )  $\text{\AA}$ ] with C(5) and C(6) out of this plane and on opposite sides [ $0.277$  ( $4$ ) and  $-0.515$  ( $3$ )  $\text{\AA}$  respectively]. The cyclohexane ring has a chair con-

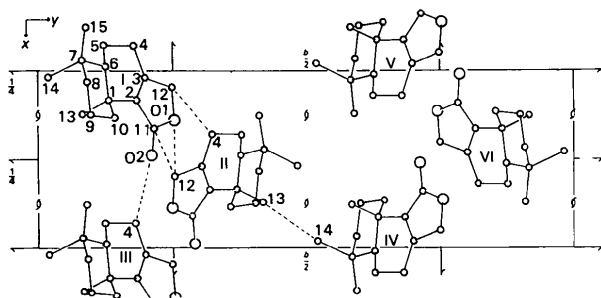


Fig. 2. The crystal structure viewed along the *c* axis. H atoms omitted.

formation: atoms C(7) and C(10) deviate +0.639 (3) and -0.695 (4) Å, respectively, from the mean plane of the other four atoms [maximum deviation 0.001 (5) Å]. The distance between the axial methyl groups, C(13)···C(14) = 3.250 (7) Å, is even shorter than the corresponding one in drimenol: 3.359 (6) Å [average of three molecules; Escobar & Wittke (1984)] and also than that in a new derivative of the latter: 3.29 (1) Å (Brianso, Piniella, Germain & Solans, 1987). Closely related to isodrimenin is alantolactone (the lactone ring is *cis* fused to the cyclohexene ring) where the distance under discussion is 3.44 (1) Å, as calculated from the published coordinates (Schmalle, 1986). Among other compounds where this steric arrangement of the cyclohexane methyl groups is observed are forskolin with C···C 3.23 (1) Å (Paulus, 1980) and an *ent*-beyerene diterpene with C···C 3.245 (8) Å (Watson, Tavanaiepour, Lee & Mabry, 1986). Bond lengths and bond angles in the present structure are comparable to those observed in the compounds referenced above.

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## Structure of 2-Amino-3-methyl-5-(*D*-arabino-1,2,3,4-tetrahydroxybutyl)imidazolium Chloride

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**Abstract.**  $C_8H_{16}N_3O_4^+Cl^-$ ,  $M_r = 253.7$ , orthorhombic,  $P2_12_12_1$ ,  $a = 10.289$  (5),  $b = 15.343$  (8),  $c = 7.367$  (4) Å,  $V = 1163.0$  (6) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.45$ ,  $D_x = 1.45$  Mg m<sup>-3</sup>,  $\lambda(Mo K\alpha) = 0.71069$  Å,  $\mu = 0.32$  mm<sup>-1</sup>,  $F(000) = 536$ , room temperature, final  $R = 0.08$  for 1372 observed reflections. The imidazole group is involved in a protonation process. The delocalization of the positive charge was evidenced by

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A view of the crystal packing is given in Fig. 2, where the dashed lines show the most significant intermolecular proximities, as given in Table 2.

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MNDO calculations. The configuration in the crystals and in the isolated molecule is biplanar (angles between planes 7.1°); also the configuration around the chiral centres C(8), C(7) and C(6) of the sugar chain is *R*, *R* and *S*, *i.e.* *D*-arabino. Packing is governed by van der Waals forces and hydrogen bonds. The Cl anion is involved in two H bonds, one with the sugar and the other with the imino group.

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