L'accès en synthèse à ce type de structure hétéro-
cyclique peu banale sera décrit dans une prochaine
publication.

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Structure of (4S,5S,7S,8S)-11-Hydroxy-1(10)-guaien-8-yl (1S)-1-Phenylethylcarbamate

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Abstract. (1) [3S-(3α,3aα,5α,6β)]-1,2,3,3a,4,5,6,7-
Octahydro-3,8-dimethyl-5-(1-hydroxy-1-methylethyl-
idene)azulen-6-yl (1S)-1-phenylethylcarbamate,
$C_{24}H_{35}NO_3$, $M_r = 385.55$, monoclinic, $P2_1$, $a =$
8.388 (2), $b = 18.520$ (5), $c = 14.132$ (3) Å, $\beta =$
98.71 (2)°, $V = 2170$ (2) Å^3, $Z = 4$, $D_x =$
1.180 (2) g cm^{-3}, $\lambda(Mo\ K\alpha) = 0.71073$ Å, $\mu =$
0.715 cm^{-1}, $F(000) = 840$, $T = 110$ K, final $R = 0.051$
for 3360 unique observed reflections. The asymmetric
unit contains molecules of approximately the same
conformation, only the arrangement of the phenyl
rings is slightly different. An intramolecular hydro-
gen bond is observed in both molecules, O(16)—
O(19) = 2.80 (1) Å, O(16)—H(16A)—O(19) =
165 (1)° and O(16')—O(19') = 2.84 (1) Å, O(16')—
H(16')—O(19') = 172 (1)°. The cycloheptene moiety
adopts a twisted conformation which is a low energy
conformer according to Allinger & Sprague [*J. Am.
Chem. Soc.* (1972), **94**, 5734–5747]. The cyclopentane
moiety adopts a slightly distorted envelope confor-
mation. A search for CC(C)OC(=O)NC(C)C frag-
ments in the Cambridge Structural Database (release
of July, 1989), giving a total of 82 molecules, showed
a planar arrangement of the urethane fragments
[OC(=O)N], the arrangement being *trans-trans* in 75
molecules and *trans-cis* in 7 molecules. The *trans-
trans* arrangement is also favoured in the present
compound.

Experimental. (I) was prepared from (4S,5S,7S,8S)-
1(10)-guaien-8,11-diol and (S)-(−)-1-phenylethyliso-
cyanate (Lemmich, Smitt, Jensen & Christensen
(1991). Crystallization from ether–petroleum ether
gave colourless plates, m.p. 401–402 K. $[\alpha]_D^{20\ C}$
+11.2°. Crystal dimensions 0.1 × 0.3 × 0.4 mm.
Preliminary film data indicated the space groups $P2_1$
or $P2_1/m$, with the reflections *hkl*: $h + l = 2n + 1$
systematically weak but observable. $P2_1$ was chosen
since the crystals are chiral. Diffraction data were
collected on an Enraf–Nonius CAD-4 diffractom-
eter. The crystal was cooled to 110 ± 0.8 K in a
stream of N_2 gas. Unit-cell dimensions were
determined from a least-squares fit of 25 reflections,
well distributed in reciprocal space, $15 < \theta < 21°$.
7518 reflections were measured with $\omega/2\theta$ scans,
$2\theta \leq 50.0°$, *hkl* ranges: *h*: 0→9, *k*: −21→21,
l: −16→16. Three reflections (10$\bar{3}$, 032 and 020)
were measured every 2.8 h to check for decay of the
crystal, which decay was not observed. The intensity
data were corrected for Lorentz–polarization effects
but not for absorption. They were averaged to 6078
independent reflections [$R_{int}(I) = 0.034$], 3360 of
which with $F_o^2 > 3.0\sigma(F_o^2)$ were considered observed.
The structure was solved by direct methods using
SHELXS86 (Sheldrick, 1990) and least-squares
refined on F with the *SDP* package (Enraf–Nonius,
1981). The H atoms were located and included in

Table 1. *Fractional atomic coordinates and isotropic thermal parameters* (Å^2)

Anisotropically refined displacement parameters are given in the isotropic equivalent form defined as: $B_{eq} = (4/3)[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + (2ab\cos\gamma)\beta_{12} + (2ac\cos\beta)\beta_{13} + (2bc\cos\alpha)\beta_{23}]$.

	x	y	z	B_{eq}/B_{iso}
C(1)	0·4025 (6)	0·1445	0·5904 (3)	1·29 (9)
C(2)	0·4113 (7)	0·1085 (3)	0·4948 (3)	1·7 (1)
C(3)	0·5212 (7)	0·1588 (3)	0·4472 (4)	2·8 (1)
C(4)	0·6375 (7)	0·1905 (3)	0·5316 (4)	1·7 (1)
C(5)	0·5174 (6)	0·2075 (3)	0·6024 (3)	1·43 (9)
C(6)	0·6024 (6)	0·2208 (2)	0·7058 (3)	1·26 (9)
C(7)	0·5043 (6)	0·2630 (3)	0·7683 (3)	1·43 (9)
C(8)	0·3305 (6)	0·2342 (2)	0·7688 (3)	1·06 (9)
C(9)	0·2974 (6)	0·1544 (2)	0·7480 (3)	1·34 (9)
C(10)	0·3107 (6)	0·1204 (3)	0·6514 (3)	1·22 (9)
C(15)	0·7672 (7)	0·1377 (3)	0·5705 (4)	2·3 (1)
C(11)	0·5957 (6)	0·2727 (3)	0·8720 (3)	1·5 (1)
C(12)	0·6103 (7)	0·2032 (3)	0·9305 (4)	1·9 (1)
C(13)	0·7604 (7)	0·3057 (3)	0·8735 (4)	2·1 (1)
O(16)	0·5030 (4)	0·3213 (2)	0·9238 (2)	2·06 (8)
O(17)	0·2178 (4)	0·2742 (2)	0·6981 (2)	1·34 (7)
C(18)	0·1763 (6)	0·3406 (2)	0·7251 (3)	1·41 (9)
O(19)	0·2238 (4)	0·3663 (2)	0·8045 (2)	1·55 (7)
N(20)	0·0795 (5)	0·3744 (2)	0·6540 (3)	1·25 (7)
C(21)	0·0198 (6)	0·4477 (2)	0·6667 (3)	1·24 (9)
C(22)	0·0595 (7)	0·4952 (3)	0·5863 (3)	1·9 (1)
C(23)	−0·1582 (6)	0·4493 (3)	0·6718 (3)	1·6 (1)
C(24)	−0·2692 (7)	0·4134 (3)	0·6044 (4)	2·0 (1)
C(25)	−0·4329 (7)	0·4178 (3)	0·6101 (4)	2·3 (1)
C(26)	−0·4864 (7)	0·4570 (3)	0·6801 (4)	2·5 (1)
C(27)	−0·3799 (7)	0·4935 (3)	0·7475 (4)	2·2 (1)
C(28)	−0·2162 (6)	0·4898 (3)	0·7425 (3)	1·8 (1)
C(14)	0·2055 (7)	0·0547 (3)	0·6333 (4)	1·9 (1)
C(1')	0·0211 (6)	0·6686 (2)	0·8889 (3)	1·28 (9)
C(2')	−0·0260 (7)	0·6335 (3)	0·9780 (4)	2·3 (1)
C(3')	−0·1446 (7)	0·6847 (3)	1·0113 (4)	2·2 (1)
C(4')	−0·2242 (6)	0·7247 (3)	0·9214 (3)	1·6 (1)
C(5')	−0·0793 (6)	0·7370 (2)	0·8679 (3)	1·31 (9)
C(6')	−0·1295 (6)	0·7543 (2)	0·7611 (3)	1·22 (9)
C(7')	0·0014 (6)	0·7924 (2)	0·7153 (3)	1·22 (9)
C(8')	0·1681 (6)	0·7574 (2)	0·7320 (3)	1·11 (9)
C(9')	0·1782 (6)	0·6749 (2)	0·7488 (3)	1·27 (9)
C(10')	0·1276 (6)	0·6424 (2)	0·8384 (3)	1·28 (9)
C(15')	−0·3557 (7)	0·6773 (3)	0·8643 (4)	2·2 (1)
C(11')	−0·0566 (7)	0·8059 (3)	0·6063 (3)	1·7 (1)
C(12')	−0·0709 (6)	0·7376 (3)	0·5479 (3)	1·7 (1)
C(13')	−0·2137 (7)	0·8483 (3)	0·5902 (4)	2·4 (1)
O(16')	0·0625 (4)	0·8507 (2)	0·5682 (2)	2·20 (8)
O(17')	0·2660 (4)	0·7889 (2)	0·8171 (2)	1·16 (7)
C(18')	0·3223 (6)	0·8560 (3)	0·8019 (3)	1·56 (9)
O(19')	0·3009 (4)	0·8870 (2)	0·7254 (2)	1·78 (8)
N(20')	0·3960 (5)	0·8848 (2)	0·8872 (3)	1·82 (8)
C(21')	0·4577 (6)	0·9589 (2)	0·8867 (3)	1·4 (1)
C(22')	0·4233 (6)	0·9948 (3)	0·9792 (3)	1·9 (1)
C(23')	0·6350 (6)	0·9625 (3)	0·8762 (3)	1·44 (9)
C(24')	0·7531 (7)	0·9315 (3)	0·9434 (3)	1·8 (1)
C(25')	0·9159 (7)	0·9361 (3)	0·9314 (4)	2·0 (1)
C(26')	0·9620 (7)	0·9725 (3)	0·8556 (4)	2·5 (1)
C(27')	0·8440 (7)	1·0050 (3)	0·7896 (4)	2·3 (1)
C(28')	0·6817 (7)	0·9990 (3)	0·7988 (4)	2·3 (1)
C(14')	0·2209 (7)	0·5736 (3)	0·8684 (4)	2·0 (1)

Fourier summations. 255 parameters were refined to $R = 0·051$, $wR = 0·049$ and $S = 1·24$ with $w = [\sigma^2(F) + 0·005F^2]^{-1}$; max. and min. heights in final $\Delta\rho$ map are 0·59 (3) and −0·45 (3) e Å^{-3}, $(\Delta/\sigma)_{max} = 0·003$. Atomic scattering factors were taken from *International Tables for Crystallography* (1974, Vol. IV, Table 2.2B), except for H atoms for which they were taken from Stewart, Davidson & Simpson (1965). The number of observed reflections allowed for anisotropic refinement of the oxygen but not the other atoms.

The asymmetric unit contains two molecules of the title compound. In one of the molecules two positions for the hydroxylic H(16) atom are located in

Table 2. *Selected bond distances* (Å) *and angles* ($°$)

	Unprimed molecule	Primed molecule
C(1)—C(2)	1·518 (6)	1·523 (7)
C(1)—C(5)	1·507 (6)	1·524 (7)
C(1)—C(10)	1·318 (7)	1·318 (7)
C(2)—C(3)	1·536 (8)	1·501 (8)
C(3)—C(4)	1·539 (7)	1·533 (7)
C(4)—C(5)	1·556 (8)	1·543 (8)
C(4)—C(15)	1·504 (7)	1·539 (7)
C(5)—C(6)	1·546 (6)	1·538 (6)
C(6)—C(7)	1·513 (7)	1·528 (7)
C(7)—C(8)	1·552 (7)	1·527 (7)
C(7)—C(11)	1·558 (6)	1·565 (6)
C(8)—C(9)	1·525 (6)	1·547 (6)
C(8)—O(17)	1·468 (5)	1·470 (5)
C(9)—C(10)	1·523 (7)	1·521 (7)
C(10)—C(14)	1·501 (7)	1·520 (7)
C(11)—C(12)	1·525 (7)	1·505 (7)
C(11)—C(13)	1·509 (8)	1·520 (8)
C(11)—O(16)	1·457 (6)	1·462 (6)
O(17)—C(18)	1·349 (6)	1·358 (6)
C(18)—O(19)	1·228 (5)	1·213 (5)
C(18)—N(20)	1·347 (6)	1·377 (6)
N(20)—C(21)	1·468 (6)	1·466 (6)
C(21)—C(22)	1·512 (7)	1·532 (7)
C(21)—C(23)	1·506 (8)	1·519 (8)
C(23)—C(24)	1·395 (7)	1·389 (7)
C(23)—C(28)	1·395 (7)	1·391 (7)
C(24)—C(25)	1·390 (8)	1·405 (8)
C(25)—C(26)	1·356 (8)	1·370 (8)
C(26)—C(27)	1·380 (7)	1·391 (7)
C(27)—C(28)	1·387 (8)	1·391 (8)
C(2)—C(1)—C(5)	108·9 (4)	108·1 (4)
C(2)—C(1)—C(10)	123·0 (3)	124·9 (4)
C(5)—C(1)—C(10)	128·0 (4)	126·9 (4)
C(1)—C(2)—C(3)	103·7 (4)	104·9 (4)
C(2)—C(3)—C(4)	104·2 (4)	105·8 (4)
C(3)—C(4)—C(5)	100·4 (4)	101·6 (4)
C(3)—C(4)—C(15)	111·9 (4)	110·2 (4)
C(5)—C(4)—C(15)	113·6 (4)	112·5 (4)
C(1)—C(5)—C(4)	103·7 (4)	103·6 (4)
C(1)—C(5)—C(6)	115·2 (4)	115·2 (4)
C(4)—C(5)—C(6)	112·9 (4)	113·2 (4)
C(5)—C(6)—C(7)	114·9 (4)	113·9 (4)
C(6)—C(7)—C(8)	114·9 (4)	116·0 (4)
C(6)—C(7)—C(11)	112·0 (4)	110·7 (4)
C(8)—C(7)—C(11)	111·3 (4)	111·2 (4)
C(7)—C(8)—C(9)	118·4 (4)	118·1 (4)
C(7)—C(8)—O(17)	109·4 (4)	110·1 (4)
C(9)—C(8)—O(17)	106·1 (3)	104·8 (3)
C(8)—C(9)—C(10)	122·6 (4)	120·2 (4)
C(1)—C(10)—C(9)	125·1 (4)	127·0 (4)
C(1)—C(10)—C(14)	123·4 (4)	121·4 (4)
C(9)—C(10)—C(14)	111·5 (4)	111·5 (4)
C(7)—C(11)—C(12)	113·6 (4)	113·1 (4)
C(7)—C(11)—C(13)	112·3 (4)	111·3 (4)
C(7)—C(11)—O(16)	108·7 (4)	109·1 (4)
C(12)—C(11)—C(13)	109·7 (4)	110·9 (4)
C(12)—C(11)—O(16)	104·7 (4)	106·0 (4)
C(13)—C(11)—O(16)	107·4 (4)	106·2 (4)
C(8)—O(17)—C(18)	115·7 (3)	113·4 (3)
O(17)—C(18)—O(19)	123·5 (4)	124·5 (4)
O(17)—C(18)—N(20)	111·4 (4)	109·7 (4)
O(19)—C(18)—N(20)	125·2 (4)	125·6 (4)
C(18)—N(20)—C(21)	121·2 (4)	118·1 (4)
N(20)—C(21)—C(22)	109·2 (4)	107·0 (4)
N(20)—C(21)—C(23)	112·5 (4)	113·2 (4)
C(22)—C(21)—C(23)	111·0 (4)	112·2 (4)
C(21)—C(23)—C(24)	121·4 (5)	121·4 (4)
C(21)—C(23)—C(28)	120·1 (4)	119·7 (4)
C(24)—C(23)—C(28)	118·4 (5)	118·9 (5)
C(23)—C(24)—C(25)	119·7 (5)	119·8 (5)
C(24)—C(25)—C(26)	120·8 (5)	121·3 (5)
C(25)—C(26)—C(27)	121·0 (6)	118·7 (5)
C(26)—C(27)—C(28)	118·9 (5)	120·6 (5)
C(23)—C(28)—C(27)	121·2 (4)	120·5 (5)

the difference Fourier map. Both of these positions were included with a population parameter of 0·5. The two positions of H(16) can be explained by the possibility of a hydrogen bond being formed either intramolecularly to O(19) or intermolecularly to
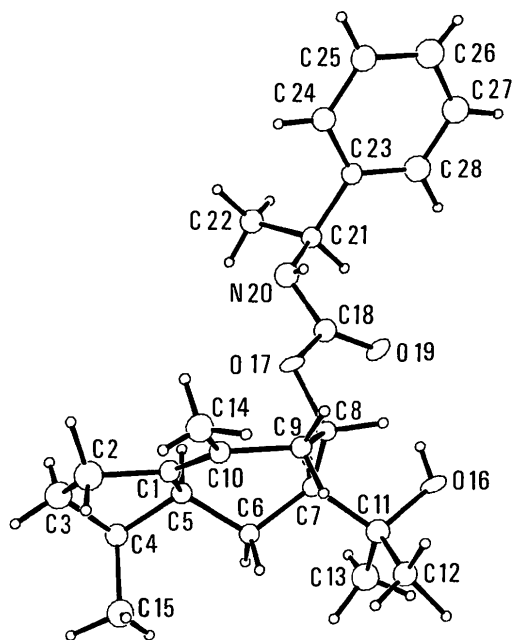
Fig. 1. *ORTEP* (Johnson, 1976) drawing of (4*S*,5*S*,7*S*,8*S*)-11-hydroxy-1(10)-guaien-8-yl (1*S*)-1-phenylethylcarbamate. The thermal ellipsoids for non-H atoms are drawn at 50% probability level.

and angles are given in Table 2. A perspective drawing of one of the molecules is shown in Fig. 1 giving the atomic numbering scheme.

Related literature. In a chemotaxonomic study of the genus *Thapsia* the distribution of sesquiterpenes and sesquiterpene lactones was investigated (Smitt, 1991). Within the species *Thapsia villosa var.* minor morphological as well as phytochemical differences were revealed. The occurrence of three new compounds, which are monoesters of a sesquiterpene diol with a guaian skeleton, in only some specimens is of chemotaxonomic value (Lemmich, Smitt, Jensen & Christensen, 1991). The structure of the three compounds was elucidated by spectroscopic methods, but it was not possible to establish the relative configuration at all the chiral centres in the sesquiterpene moiety from the NMR spectroscopic data.

N(20'). Final atomic coordinates and isotropic or equivalent isotropic displacement parameters of non-H atoms are listed in Table 1.* Bond distances

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Structure of a Model for Aspirochlorine (Antibiotic A30641)

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Abstract. (1) $C_{20}H_{18}N_2O_5S_2$, $M_r = 430.48$, monoclinic, $P2_1/c$, $a = 9.934$ (3), $b = 18.730$ (5), $c = 10.563$ (2) Å, $\beta = 96.23$ (2)°, $V = 1954$ (1) Å^3, $Z = 4$, $D_x = 1.46$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 3.0$ cm^{-1}, $F(000) = 896$, $T = 112$ K, $R = 0.046$ ($wR = 0.041$) for 2813 unique, observed reflections. The compound is a model for aspirochlorine (antibiotic A30641).

H₃CO

OCH₃

(1)