cembrane class. Their presence is of interest, since these carbomonocyclic diterpenoids are prone to undergo biodegradation thereby giving rise to odoriferous carboacyclic products (Wahlberg & Enzell, 1987). It is also noteworthy that some tobacco cembranoids have biological functions, *e.g.* as growth inhibitors (Springer, Clardy, Cox, Cutler & Cole, 1975) and insect repellents (Johnson & Severson, 1984) and that several exhibit antitumour-promoter effects (Saito, Tsujino, Kaneko, Yoshida & Mizusaki, 1987).

The authors are grateful to Professors Peder Kierkegaard, Rolf Norrestam and Curt R. Enzell, and Dr Arne Björnberg for their support of this work.

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Acta Cryst. (1992). C**48**, 566–568

Structure of a 7,8-Epoxycembranoid Isolated from Tobacco

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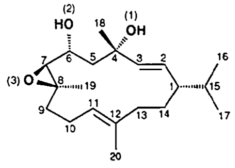
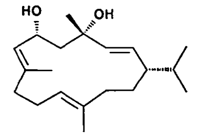
(*Received* 6 *June* 1991; *accepted* 27 *August* 1991)

Abstract. (1*S*,2*E*,4*S*,6*R*,7*S*,8*S*,11*E*)-7,8-Epoxy-2,11-cembradiene-4,6-diol monohydrate (1), $C_{20}H_{34}O_3.H_2O$, $M_r = 340.5$, monoclinic, $P2_1$, $a = 9.808$ (2), $b = 8.019$ (4), $c = 13.755$ (3) Å, $\beta = 105.73$ (2)°, $V = 1041.3$ (6) Å^3, $Z = 2$, $D_x = 1.086$ (6) g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.687$ cm^{-1}, $F(000) = 376$, $T = 293$ K, final $R = 0.063$ for 2388 unique reflections. The structure consists of a 14-membered cembrane ring, which is *trans*-fused to an epoxide function, the torsion angle C(6)—C(7)—C(8)—C(9) being −156.0 (4)°. There are two double bonds, one between C(2) and C(3) and one between C(11) and C(12). Both are of *E* geometry and have torsion angles C(1)—C(2)—C(3)—C(4) = 173.0 (4)° and C(10)—C(11)—C(12)—C(13) = −174.7 (6)°. There is one possible intramolecular hydrogen bond O(2)···O(3) = 2.921 (4) Å, O(2)—H(22)···O(3) = 109.5° and three possible intermolecular hydrogen bonds: O(1$^{\rm i}$)···O(4) = 2.839 (8), O(2$^{\rm ii}$)···O(4) = 2.989 (4) and O(4)···O(3$^{\rm ii}$) = 2.824 (5) Å, forming infinite chains in the ⟨100⟩ direction [symmetry code: (i) $-2-x, \frac{1}{2}+y, -1-z$; (ii) $-1-x, \frac{1}{2}+y, -1-z$].



(1)



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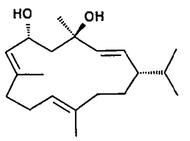

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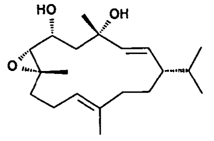

(4)

Experimental. The isolation of 3.7 mg of (1) from a chloroform extract of flowers of Greek tobacco has been described previously (Wahlberg, Eklund, Vogt, Enzell & Berg, 1986). A single crystal was obtained by recrystallization from a mixture of hexane and

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ethyl acetate. The X-ray data from this colourless prismatic crystal, size 0.32 × 0.20 × 0.72 mm, was collected with a Siemens/Stoe AED2 diffractometer using graphite-monochromated Mo $K\alpha$ radiation. The unit-cell parameters were refined from the observed θ values of 25 reflections in the range 12.5–25.0°. The symmetry and systematic extinctions of the X-ray reflections were consistent with space groups $P2_1$ or $P2_1/m$. Intensities of the X-ray reflections were measured by the ω–2θ method. A total of 4861 reflections were collected ($3 < 2\theta < 70°$), maximum $(\sin\theta)/\lambda = 0.81$ Å^{-1}, of which 2388 unique reflections were considered observed [$F > 4.0\sigma(F)$, $-15 < h < 15$, $0 < k < 12$, $0 < l < 22$]. The intensities were corrected for Lorentz and polarization effects, but no correction was made for absorption. The intensities of three standard reflections (200, 013 and $0\bar{1}3$) were monitored every hour; the total deterioration of intensity was <5%. As intensity statistics indicated a noncentric electron distribution, the space group was assumed to be $P2_1$. This space group symmetry was supported by the outcome of the structure refinements.

The positions of 22 of the 24 non-H atoms were obtained by direct methods using the *MULTAN*80 program package (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), while the remaining non-H atoms and the two hydroxy H atoms were located from $\Delta\rho$ maps. All the non-hydroxy H atoms were geometrically placed with a distance of 1.08 Å to the adjacent C atom. The structure was refined by full-matrix least-squares techniques using the program *SHELX*76 (Sheldrick, 1976) for minimizing $\sum w(|F_o| - |F_c|)^2$. The y coordinate of the C atom C(1) was kept fixed to specify the origin along the polar axis. Anisotropic displacement parameters were used for all non-H atoms, while all H atoms were refined with an isotropic displacement factor constrained to 0.05 Å^2. The number of refined parameters was 244. Final agreement factors: $R = 0.063$, $wR = 0.070$ ($wR = 0.099$ for all significant reflections) using the weighting scheme $w = [\sigma^2(F) + g|F|^2]^{-1}$ with $g = 0.0030$. $(\Delta/\sigma)_{\max} = 0.12$, $\Delta\rho_{\min} = -0.11$ and $\Delta\rho_{\max} = 0.12$ e Å^{-3}. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were carried out on a C220 Convex computer.

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement coefficients* (Å × 10^2)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	−8136 (4)	526	−1927 (3)	4.26 (11)
C(2)	−8329 (4)	25 (7)	−3005 (3)	3.90 (10)
C(3)	−7442 (3)	−903 (7)	−3348 (2)	3.16 (8)
C(4)	−7699 (3)	−1568 (7)	−4410 (2)	2.97 (8)
C(5)	−6558 (3)	−1069 (7)	−4931 (2)	3.01 (8)
C(6)	−5049 (3)	−1742 (7)	−4527 (2)	2.80 (8)
C(7)	−4200 (3)	−774 (7)	−3623 (2)	3.12 (9)
C(8)	−3632 (4)	−1476 (8)	−2596 (3)	3.92 (10)
C(9)	−3306 (4)	−358 (9)	−1668 (3)	5.32 (13)
C(10)	−3296 (5)	1529 (9)	−1834 (4)	6.11 (16)
C(11)	−4748 (5)	2257 (8)	−2202 (3)	5.61 (14)
C(12)	−5567 (5)	2821 (8)	−1648 (3)	5.42 (14)
C(13)	−7066 (7)	3382 (8)	−2162 (4)	6.75 (18)
C(14)	−8219 (5)	2428 (9)	−1831 (4)	6.08 (17)
C(15)	−9243 (5)	−403 (9)	−1496 (3)	5.44 (14)
C(16)	−9141 (7)	−2255 (10)	−1585 (4)	7.22 (19)
C(17)	−9097 (7)	67 (12)	−392 (4)	8.0 (20)
C(18)	−7852 (4)	−3473 (7)	−4397 (3)	4.22 (10)
C(19)	−3840 (6)	−3294 (9)	−2374 (4)	6.23 (17)
C(20)	−5115 (7)	2934 (11)	−514 (4)	8.3 (3)
O(1)	−9027 (2)	−969 (6)	−5040 (2)	3.58 (7)
O(2)	−4414 (3)	−1476 (7)	−5346 (2)	3.93 (8)
O(3)	−2715 (2)	−1218 (7)	−3252 (2)	4.78 (8)
O(4)	−8619 (3)	2484 (7)	−5332 (2)	4.45 (8)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *for the non-*H *atoms*

C(2)—C(1)	1.498 (6)	C(14)—C(1)	1.535 (9)
C(15)—C(1)	1.560 (8)	C(3)—C(2)	1.325 (5)
C(4)—C(3)	1.512 (6)	C(5)—C(4)	1.537 (6)
C(18)—C(4)	1.535 (6)	O(1)—C(4)	1.438 (5)
C(6)—C(5)	1.531 (6)	C(7)—C(6)	1.510 (6)
O(2)—C(6)	1.443 (4)	C(8)—C(7)	1.482 (6)
O(3)—C(7)	1.451 (5)	C(9)—C(8)	1.521 (8)
C(19)—C(8)	1.515 (8)	O(3)—C(8)	1.450 (5)
C(10)—C(9)	1.531 (10)	C(11)—C(10)	1.496 (8)
C(12)—C(11)	1.327 (7)	C(13)—C(12)	1.517 (10)
C(20)—C(12)	1.504 (8)	C(14)—C(13)	1.533 (10)
C(16)—C(15)	1.496 (10)	C(17)—C(15)	1.533 (8)

C(14)—C(1)—C(2)	110.8 (5)	C(15)—C(1)—C(2)	109.5 (4)
C(15)—C(1)—C(14)	112.5 (4)	C(3)—C(2)—C(1)	125.7 (4)
C(4)—C(3)—C(2)	126.1 (4)	C(5)—C(4)—C(3)	114.0 (3)
C(18)—C(4)—C(3)	109.3 (4)	C(18)—C(4)—C(5)	110.5 (4)
O(1)—C(4)—C(3)	110.6 (3)	O(1)—C(4)—C(5)	106.7 (3)
O(1)—C(4)—C(18)	105.3 (4)	C(6)—C(5)—C(4)	119.6 (3)
C(7)—C(6)—C(5)	112.4 (4)	O(2)—C(6)—C(5)	104.1 (3)
O(2)—C(6)—C(7)	107.9 (3)	C(8)—C(7)—C(6)	124.7 (4)
O(3)—C(7)—C(6)	116.1 (4)	O(3)—C(7)—C(8)	59.3 (3)
C(9)—C(8)—C(7)	121.0 (5)	C(19)—C(8)—C(7)	121.5 (4)
C(19)—C(8)—C(9)	114.0 (5)	O(3)—C(8)—C(7)	59.3 (3)
O(3)—C(8)—C(9)	114.4 (4)	O(3)—C(8)—C(19)	113.8 (5)
C(10)—C(9)—C(8)	117.8 (5)	C(11)—C(10)—C(9)	113.1 (5)
C(12)—C(11)—C(10)	127.4 (5)	C(13)—C(12)—C(11)	119.6 (5)
C(20)—C(12)—C(11)	124.2 (6)	C(20)—C(12)—C(13)	116.2 (5)
C(14)—C(13)—C(12)	114.4 (5)	C(13)—C(14)—C(1)	114.2 (5)
C(16)—C(15)—C(1)	112.0 (5)	C(17)—C(15)—C(1)	111.8 (5)
C(17)—C(15)—C(16)	109.5 (6)	C(8)—O(3)—C(7)	61.4 (3)

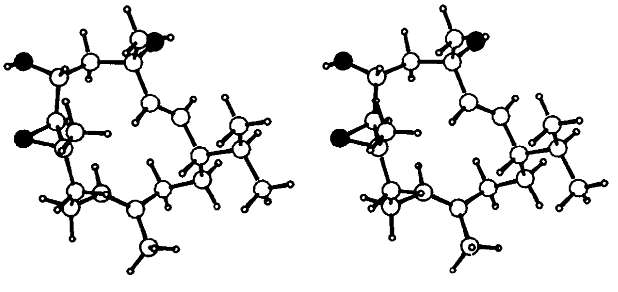

Fig. 1. A stereoscopic view of (1*S*,2*E*,4*S*,6*R*,7*S*,8*S*,11*E*)-7,8-epoxy-2,11-cembradiene-4,6-diol (1). The O atoms are represented as filled circles.

The atom-numbering scheme of the 7,8-epoxide (1) is given in the scheme above [the O atom of the crystal water is designated O(4)]. A *PLUTO* (Motherwell & Clegg, 1978) stereo drawing of the molecule is shown in Fig. 1. The final fractional atomic coordinates with standard deviations and equivalent isotropic temperature factors for the non-H atoms are listed in Table 1.* Bond lengths and bond angles, both with e.s.d.'s, are given in Table 2.

Related literature. Some seventy diterpenoids of the cembrane class have hitherto been reported as constituents of tobacco. These compounds are present in the cuticular wax of the leaf and flower and include as the major components the (4*S*,6*R*)- and (4*R*,6*R*)-diols (2) and (3) (Wahlberg & Enzell, 1987). The (7*S*,8*S*)-epoxide (1) occurs along with the corresponding (7*R*,8*R*)-epoxide (4) in tobacco. Both epoxides are plausible metabolites of the (4*S*,6*R*)-diol (2), and their absolute configurations have been determined by chemical correlation with this diol (Wahlberg, Eklund, Vogt, Enzell & Berg, 1986).

The authors are grateful to Professors Peder Kierkegaard, Rolf Norrestam and Curt R. Enzell and Dr Arne Björnberg for their support of this work.

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* Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54636 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0228]

Acta Cryst. (1992). C**48**, 568–570

Structure of *trans*-3,3-Dichloro-4-(α-chlorobenzyl)-1-methyl-5-phenyl-2-pyrrolidinone

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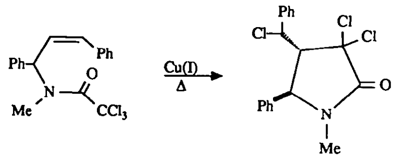
(*Received* 28 *June* 1991; *accepted* 11 *July* 1991)

Abstract. $C_{18}H_{16}Cl_3NO$, $M_r = 368.69$, triclinic, $P\bar{1}$, $a = 10.360\,(1)$, $b = 10.397\,(1)$, $c = 10.810\,(2)$ Å, $\alpha = 60.84\,(1)$, $\beta = 57.22\,(1)$, $\gamma = 70.97\,(1)°$, $V = 852\,(1)$ Å^3, $Z = 2$, $D_x = 1.437$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu = 0.54$ mm^{-1}, $F(000) = 380$, $T = 293\,(1)$ K, $R = 0.0287$ for 2330 observed reflections with $I > 3\sigma(I)$. The five-membered ring has an envelope conformation and the 4-(α-chlorobenzyl) and 5-phenyl groups are *trans* with respect to each other.

Experimental. The title compound (2) was synthesized by radical cyclization of the allylic amide (1) which occurred in a completely regio- and stereoselective manner in excellent yield.



(1) (2)

A colorless prismatic crystal of approximate dimensions 0.47 × 0.60 × 0.50 mm was used for data collection. Cell parameters and crystal orientation matrix were determined on an Enraf–Nonius CAD-4 diffractometer by a least-squares refinement of the setting angles of 25 reflections with $10 < \theta < 15°$. Intensity data were collected by the $\omega/2\theta$ scan

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0108-2701/92/030568-03$03.00