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 (4S,6R)- and (4R,6R)-diols (2) and (3) (Wahlberg & Enzell, 1987). The (7S,8S)-epoxide (1) occurs along with the corresponding (7R,8R)-epoxide (4) in tobacco. Both epoxides are plausible metabolites of the (4S,6R)-diol

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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]

(2), and their absolute configurations have been determined by chemical correlation with this diol (Wahlberg, Eklund, Vogt, Enzell & Berg, 1986).

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

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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)

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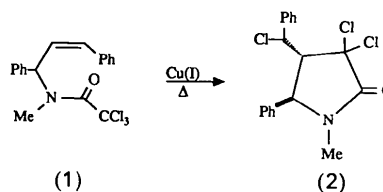
A colorless prismatic crystal of approximate dimensions 0.47 × 0.60 × 0.50 mm was used for data collection. Cell parameters and crystal orientation matrix were determined on an Enraf–Nonius CAD-4 diffractometer by a least-squares refinement of the setting angles of 25 reflections with $10 < \theta <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

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters* ($Å^2$) *with e.s.d.'s in parentheses*

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

	x	y	z	B_{eq}
Cl(1)	0.57055 (6)	0.10215 (6)	0.58387 (6)	4.33 (2)
Cl(2)	0.90053 (6)	0.09719 (7)	0.39821 (6)	4.61 (2)
Cl(3)	0.96948 (6)	0.32836 (6)	0.00685 (7)	4.57 (2)
O	0.7490 (2)	0.3958 (2)	0.4256 (2)	4.45 (4)
N	0.6415 (2)	0.4286 (2)	0.2741 (2)	2.86 (4)
C(1)	0.6076 (2)	0.5870 (2)	0.2246 (3)	4.07 (6)
C(2)	0.7103 (2)	0.3503 (2)	0.3677 (2)	3.04 (5)
C(3)	0.7272 (2)	0.1889 (2)	0.3919 (2)	2.99 (5)
C(4)	0.7056 (2)	0.1945 (2)	0.2593 (2)	2.56 (5)
C(5)	0.6136 (2)	0.3466 (2)	0.2140 (2)	2.51 (5)
C(6)	0.8540 (2)	0.1755 (2)	0.1145 (2)	2.92 (5)
C(51)	0.4438 (2)	0.3388 (2)	0.2767 (2)	2.68 (5)
C(52)	0.3308 (2)	0.3577 (2)	0.4153 (2)	3.75 (6)
C(53)	0.1774 (3)	0.3484 (3)	0.4679 (3)	4.60 (7)
C(54)	0.1366 (2)	0.3202 (3)	0.3832 (3)	4.47 (7)
C(55)	0.2480 (2)	0.3007 (3)	0.2457 (2)	4.29 (6)
C(56)	0.4011 (2)	0.3110 (2)	0.1909 (2)	3.52 (5)
C(61)	0.8174 (2)	0.1531 (2)	0.0089 (2)	2.86 (5)
C(62)	0.8166 (2)	0.2625 (2)	−0.1308 (2)	3.76 (6)
C(63)	0.7742 (3)	0.2343 (3)	−0.2158 (2)	4.63 (7)
C(64)	0.7306 (3)	0.0984 (3)	−0.1615 (2)	5.10 (7)
C(65)	0.2689 (3)	0.0114 (3)	0.0228 (3)	4.90 (7)
C(66)	0.7753 (2)	0.0153 (2)	0.0611 (2)	3.65 (6)

Table 2. *Bond distances* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Cl(1)—C(3)	1.799 (2)	C(51)—C(52)	1.375 (2)
Cl(2)—C(3)	1.759 (1)	C(51)—C(56)	1.389 (2)
Cl(3)—C(6)	1.800 (2)	C(52)—C(53)	1.388 (2)
O—C(2)	1.208 (2)	C(53)—C(54)	1.366 (3)
N—C(1)	1.446 (2)	C(54)—C(55)	1.366 (3)
N—C(2)	1.340 (2)	C(55)—C(56)	1.382 (2)
N—C(5)	1.461 (2)	C(61)—C(62)	1.381 (2)
C(2)—C(3)	1.531 (2)	C(61)—C(66)	1.384 (2)
C(3)—C(4)	1.538 (2)	C(62)—C(63)	1.381 (3)
C(4)—C(5)	1.555 (2)	C(63)—C(64)	1.365 (3)
C(4)—C(6)	1.533 (2)	C(64)—C(65)	1.374 (3)
C(5)—C(51)	1.520 (2)	C(65)—C(66)	1.374 (3)
C(6)—C(61)	1.512 (3)		

C(1)—N—C(2)	122.2 (1)	Cl(2)—C(3)—C(4)	116.0 (1)
C(1)—N—C(5)	121.6 (1)	C(2)—C(3)—C(4)	105.4 (1)
C(2)—N—C(5)	116.1 (1)	C(3)—C(4)—C(5)	103.4 (1)
O—C(2)—N	127.4 (2)	C(3)—C(4)—C(6)	115.7 (1)
O—C(2)—C(3)	126.0 (2)	C(5)—C(4)—C(6)	112.6 (1)
N—C(2)—C(3)	106.6 (1)	N—C(5)—C(4)	103.4 (1)
Cl(1)—C(3)—Cl(2)	107.9 (1)	N—C(5)—C(51)	113.7 (1)
Cl(1)—C(3)—C(2)	106.1 (1)	C(4)—C(5)—C(51)	114.8 (1)
Cl(1)—C(3)—C(4)	109.7 (1)	Cl(3)—C(6)—C(4)	110.9 (1)
Cl(2)—C(3)—C(2)	111.3 (1)	Cl(3)—C(6)—C(61)	112.3 (1)
C(4)—C(6)—C(61)	110.6 (1)	C(6)—C(61)—C(62)	124.3 (2)
C(5)—C(51)—C(52)	122.6 (1)	C(6)—C(61)—C(66)	117.1 (1)
C(5)—C(51)—C(56)	118.7 (1)	C(62)—C(61)—C(66)	118.6 (2)
C(52)—C(51)—C(56)	118.7 (2)	C(61)—C(62)—C(63)	120.4 (2)
C(51)—C(52)—C(53)	120.3 (2)	C(62)—C(63)—C(64)	120.3 (2)
C(52)—C(53)—C(54)	120.6 (2)	C(63)—C(64)—C(65)	119.9 (2)
C(53)—C(54)—C(55)	119.5 (2)	C(64)—C(65)—C(66)	120.1 (2)
C(54)—C(55)—C(56)	120.6 (2)	C(61)—C(66)—C(65)	120.7 (2)
C(51)—C(56)—C(55)	120.3 (2)		

method using variable scan speed (0.52–3.30° min⁻¹), scan width of (0.60 + 0.35tanθ)° and graphite-monochromated radiation in the range 2 < θ < 25° with h − 12 to 12, k − 12 to 12 and l 0 to 12. Three reflections were monitored every 2 h of exposure time and showed no significant intensity variations. The intensities of 3215 unique reflections were measured, of which 2330 had $I > 3\sigma(I)$, where $\sigma^2(I) = S + 2B + [0.04(S - B)]^2$, S = scan count and B = time-averaged background count extended 25% on each side; R_f = 0.004. Data were corrected for

Lorentz, polarization and absorption (North, Phillips & Mathews, 1968) effects; the maximum and minimum absorption correction factors were 1.000 and 0.964 respectively.

The structure was solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and refined by full-matrix least-squares calculations employing F's, with anisotropic temperature factors for the non-H atoms. A difference Fourier map calculated at an intermediate stage of the refinement revealed all H atoms. These were included in the refinement with an overall isotropic temperature factor in subsequent calculations. Atomic scattering factors for non-H atoms were taken from Cromer & Mann (1968) and those for H atoms were from Stewart, Davidson & Simpson (1965), and allowance was made for anomalous dispersion (Cromer & Liberman, 1970). After several cycles of full-matrix refinement, convergence was reached with R = 0.0287 and wR = 0.0288, where $w = [(\sigma^2(F_o) + 0.050F_o)^2]^{-1}$. At the conclusion of the refinement $(\Delta/\sigma)_{max} < 0.1$, the final difference electron density map was essentially featureless with $\Delta\rho$ within ± 0.30 e Å⁻³, and goodness of fit S = 2.055.

The computer programs used in this study were from the *Enraf–Nonius Structure Determination Package* (B. A. Frenz & Associates, Inc., 1985) and *ORTEP*II (Johnson, 1976). Final fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s are given in Table 1.* Table 2 contains bond lengths and bond angles. Fig. 1 shows the molecular structure of the title compound.

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* Lists of structure amplitudes, anisotropic temperature factors, H-atom parameters and molecular dimensions involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54533 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
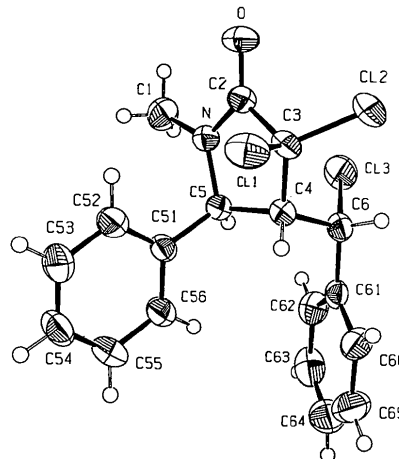

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound.

$C_{18}H_{16}Cl_3NO$

Related literature. New regio- and stereoselective preparation of trichlorinated γ-butyrolactones by copper-catalyzed cyclization of allyl trichloroacetates (Nagashima, Wakamatsu, Itoh, Tomo & Tsuji, 1983).

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Structure of 6-Fluoro-1β,2β-methylene-3,20-dioxo-9β,10α-pregna-4,6-dien-17α-yl Acetate

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Abstract. $C_{24}H_{29}FO_4$, $M_r = 400.5$, orthorhombic, $P2_12_12_1$, $a = 12.207$ (1), $b = 14.715$ (1), $c = 11.572$ (1) Å, $V = 2078.7$ (5) Å^3, $Z = 4$, $D_x = 1.28$ g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, $\mu = 7.62$ cm^{-1}, $F(000) = 856$, $T = 291$ K, final $R = 0.041$ for 2230 observed reflections. The A and B rings assume flat boat and 9β,10α-half-chair conformations, with asymmetry parameters $\Delta C_s^3 = 1.5$, $\Delta C_s^{1,2} = 11.2$ and $\Delta C_2^{6,7} = 3.1$, $\Delta C_s^7 = 15.0$, respectively.

Experimental. A crystal (0.08 × 0.40 × 0.48 mm) was grown from acetone solution. The X-ray data were collected on a CAD-4 diffractometer using Cu $K\alpha$ (Ni filter) radiation for reflections with 2θ from 4 to 150°, range h,k,l, $0 \le 15$, $0 \le 19$, $0 \le 16$; $\omega/2\theta$ scan with minimum scan speed of 4° min^{-1} in θ and scan width of $(0.75 + 0.14\tan\theta)°$. Cell dimensions calculated by least-squares refinement of 25 reflections, 50 $< 2\theta < 60°$. Intensities of two standard reflections (274, 524) were measured every 4 h and varied by less than 2% throughout the data collection. Intensities for 2478 reflections measured, 2422 unique reflections and 2230 with $F_o > 2\sigma(F_o)$ used in the refinement. The structure was determined using MULTAN78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined on F by full-matrix least-squares method. H atoms were located by difference Fourier synthesis and were refined isotropically. H atoms on C21 were unreasonable and were replaced with theoretical hydrogens, held constant. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 0.041$, $wR = 0.055$, $S = 1.555$, $w = 1/\sigma^2$, $(\Delta/\sigma)_{max} = 0.02$. Weighting scheme based on estimates of experimental errors from counting statistics. The final difference map showed maximum and minimum peaks of 0.19 and -0.20 e Å^{-3}. No corrections for absorption or extinction were made.

Atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1. The bond lengths and angles are presented in Table 2.* An

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* Lists of structure factors, anisotropic displacement parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54647 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.