between the *ac* plane and the aromatic plane is 68·7°, which is 1·2° larger than the corresponding angle of the orthorhombic phase. The increase is caused by the cancellation of the stresses arising from the enlargement of the monoclinic angle β from 90°. The x coordinate of the centre of an anilinium-ion and a bromide-ion pair is conserved at about $\frac{1}{4}$. The shortest intermolecular atomic distance is C(4)—C(6′). Next-shortest distances are C(2)—C(5′) and C(3)—C(5′). These are a little shorter than those of many other aromatic compounds.

Between consecutive layers, the strongest interactions can be expected between the Br^- ions and the NH_3^+ group. Every Br^- ion is surrounded by four NH_3^+ ions or each NH_3^+ by four Br^-. However, the distances between an N atom and the surrounding four Br^- ions are not equal to those of the high-temperature phase. One of the Br—N distances is a little longer than the other three.

When an N atom and its surrounding four Br^- ions only are taken into account, there is a local symmetry of a mirror plane perpendicular to the b axis containing the N atom and a $Br(I)^-$ ion. If the local symmetry determines the orientation of the assumed regular triangular pyramid of NH_3^+ with N—H 1·05 Å and H—N—H 109° 28′, then the distances between three H atoms and their nearest bromines are all estimated to be 2·35 Å. This distance is nearly equal to that of the sum of the H and Br ionic radii. Any other positions for the H atoms obtained by rotating the NH_3^+ group about the longest axis are unreasonable because of the interionic distances between the H atoms and Br^- ions and also because of the Coulomb potential between these ions.

The calculations were carried out on a FACOM M-190 computer at the Data Processing Center, Kyoto University. Programs for least-squares refinements and Fourier syntheses were *RSFLS*-4 and *RSSFR*-3 in *UNICS* (1967).

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Structure of 19-Acetylteuspinin, a New Clerodane Diterpenoid from the Species *Teucrium*

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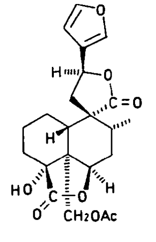
(*Received* 26 *March* 1981; *accepted* 11 *May* 1981)

Abstract. $C_{22}H_{26}O_8$, orthorhombic, $P2_12_12_1$, a = 25·569 (2), b = 13·560 (1), c = 11·993 (1) Å, U = 4158·2 (6) Å^3, M_r = 420·458, Z = 8, D_c = 1·32 Mg m^{-3}, $F(000)$ = 1776. The structure has been solved by direct methods and refined to a final R value of 6·5% for 5800 observed reflexions. Both crystallographically independent molecules are indistinguishable, showing the *ent*-clerodane-type structure. They are joined together by two H bonds, forming dimers in the crystal. The cyclohexane rings show a chair conformation and the γ-spirolactone and lactone rings have an envelope conformation. The absolute configuration is the same as that found for other molecules of the species *Teucrium*.

Introduction. From the species *Teucrium* a number of clerodane and *ent*-clerodane diterpenoids have been isolated (Piozzi, 1981). In the present work we report the crystal structure of a new clerodane, teuspinin (I) (Savona, Paternostro, Piozzi & Rodríguez, 1980).



(I)

The X-ray data were collected on an automatic four-circle diffractometer from a specimen 0·1 × 0·2 × 0·24 mm, using graphite-monochromated Cu $K\alpha$ radiation up to a Bragg angle of 65°. No intensity decay was observed. Of the 3670 Friedel pairs collected, 2900 had $I > 2\sigma(I)$ and were subsequently used for the structure determination and refinement. The data were corrected for Lorentz and polarization factors, but not for absorption. The structure was solved by *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1977), and refined by full-matrix least squares with anisotropic thermal parameters for the non-hydrogen atoms. The H atoms were fixed at idealized positions (C–H = 1·00 Å, H–C–H = 104°), checked on a difference Fourier map, and included in the refinement as fixed isotropic contributions. A convenient weighting scheme was selected to prevent bias in $\langle w\Delta^2F\rangle$ *vs* $\langle|F_o|\rangle$ and $\langle\sin\theta/\lambda\rangle$ (Martinez-Ripoll & Cano, 1975). Several cycles of weighted refinement including both hkl and $\bar{h}\bar{k}\bar{l}$ reflexions gave the unweighted and weighted discrepancy indices R = 0·065 and R_w = 0·080 for the correct enantiomer (Stewart, Kundell & Baldwin, 1970).* The absolute configuration was determined by comparing the 92 most relevant Bijvoet pairs with $F_o > 10\sigma(F_o)$, $\Delta F_c >$ 0·15 and $\Delta F_o >$ 0·05. The average Bijvoet difference for the correct enantiomer was 0·88 *vs* 0·99 for the wrong enantiomer and the averaged Bijvoet ratio was 0·073 *vs* 0·079 (Martinez-Ripoll & Fayos, 1977).

Discussion. The two independent molecules are related by a pseudo screw parallel to **b** with a translation of 0·185*b*. A drawing of the asymmetric unit showing both *ent*-clerodane molecules with their actual absolute

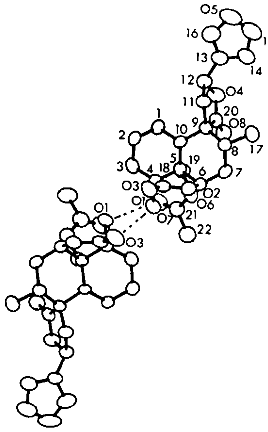


Fig. 1. The two crystallographically independent, although pseudo-symmetrically related, molecules of teuspinin, linked by two hydrogen bonds.

Table 1. *Fractional coordinates* (×10⁴) *and isotropic thermal parameters* (×10³)

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq} (Å^2)
O(1)*A*	8010 (1)	8370 (2)	1496 (2)	55 (1)
O(2)*A*	8615 (1)	10032 (2)	500 (2)	51 (1)
O(3)*A*	7798 (1)	10561 (2)	799 (3)	71 (1)
O(4)*A*	8968 (1)	8011 (2)	−3819 (2)	58 (1)
O(5)*A*	8902 (2)	9520 (4)	−7109 (3)	107 (2)
O(6)*A*	8793 (1)	7056 (2)	741 (2)	57 (1)
O(7)*A*	8102 (1)	6135 (2)	1271 (3)	79 (1)
O(8)*A*	9337 (1)	7475 (2)	−2261 (3)	61 (1)
C(1)*A*	7864 (2)	8416 (3)	−2001 (3)	51 (1)
C(2)*A*	7381 (2)	8752 (4)	−1331 (4)	68 (2)
C(3)*A*	7439 (2)	8565 (4)	−99 (4)	59 (2)
C(4)*A*	7969 (2)	8808 (3)	407 (3)	46 (1)
C(5)*A*	8444 (1)	8479 (3)	−282 (3)	40 (1)
C(6)*A*	8872 (2)	9074 (3)	285 (3)	46 (1)
C(7)*A*	9356 (2)	9255 (3)	−425 (3)	49 (1)
C(8)*A*	9251 (2)	9649 (3)	−1593 (3)	47 (1)
C(9)*A*	8838 (1)	9038 (3)	−2247 (3)	40 (1)
C(10)*A*	8347 (1)	8895 (3)	−1481 (3)	40 (1)
C(11)*A*	8673 (2)	9605 (3)	−3298 (3)	48 (1)
C(12)*A*	8647 (2)	8833 (3)	−4224 (3)	50 (1)
C(13)*A*	8848 (2)	9154 (3)	−5313 (3)	52 (1)
C(14)*A*	9361 (2)	9550 (4)	−5560 (4)	68 (2)
C(15)*A*	9365 (3)	9765 (5)	−6636 (5)	88 (2)
C(16)*A*	8588 (2)	9153 (5)	−6283 (4)	90 (2)
C(17)*A*	9772 (2)	9755 (4)	−2214 (4)	71 (2)
C(18)*A*	8093 (2)	9895 (3)	581 (3)	52 (1)
C(19)*A*	8532 (2)	7360 (3)	−269 (3)	54 (1)
C(20)*A*	9071 (2)	8090 (3)	−2712 (3)	49 (1)
C(21)*A*	8529 (2)	6474 (3)	1463 (4)	63 (2)
C(22)*A*	8843 (3)	6325 (4)	2488 (4)	90 (2)
O(1)*B*	7020 (1)	6477 (2)	2221 (3)	67 (1)
O(2)*B*	6351 (1)	8103 (2)	3046 (2)	54 (1)
O(3)*B*	7167 (1)	8687 (2)	2911 (3)	65 (1)
O(4)*B*	5823 (1)	6163 (2)	7363 (3)	71 (1)
O(5)*B*	5672 (2)	7838 (4)	10602 (3)	125 (2)
O(6)*B*	6194 (1)	5177 (2)	2853 (3)	62 (1)
O(7)*B*	6850 (1)	4134 (3)	2447 (3)	84 (1)
O(8)*B*	5588 (1)	5551 (2)	5739 (3)	67 (1)
C(1)*B*	7017 (2)	6574 (4)	5755 (4)	58 (1)
C(2)*B*	7509 (2)	6974 (4)	5167 (4)	67 (2)
C(3)*B*	7520 (2)	6719 (4)	3943 (4)	63 (2)
C(4)*B*	7010 (2)	6914 (3)	3320 (3)	48 (1)
C(5)*B*	6508 (1)	6589 (3)	3918 (3)	46 (1)
C(6)*B*	6091 (2)	7158 (3)	3273 (3)	49 (1)
C(7)*B*	5584 (2)	7315 (3)	3884 (4)	55 (1)
C(8)*B*	5641 (2)	7747 (3)	5052 (4)	50 (1)
C(9)*B*	6030 (1)	7167 (3)	5794 (3)	44 (1)
C(10)*B*	6547 (2)	7030 (3)	5122 (3)	45 (1)
C(11)*B*	6132 (2)	7754 (3)	6889 (3)	54 (1)
C(12)*B*	6111 (2)	6994 (4)	7839 (4)	62 (2)
C(13)*B*	5831 (2)	7338 (4)	8860 (4)	68 (2)
C(14)*B*	5294 (2)	7639 (5)	8954 (5)	85 (2)
C(15)*B*	5224 (3)	7921 (5)	10020 (6)	101 (3)
C(16)*B*	6037 (3)	7468 (7)	9868 (5)	109 (3)
C(17)*B*	5094 (2)	7869 (4)	5579 (4)	67 (2)
C(18)*B*	6875 (2)	8000 (3)	3085 (3)	51 (1)
C(19)*B*	6432 (2)	5450 (3)	3908 (4)	61 (2)
C(20)*B*	5794 (2)	6203 (3)	6238 (4)	55 (1)
C(21)*B*	6464 (2)	4569 (3)	2182 (4)	65 (2)
C(22)*B*	6203 (2)	4502 (4)	1059 (5)	80 (2)

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36169 (54 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

configuration is in Fig. 1. The atomic parameters are given in Table 1. A comparison of the two independent molecular geometries was carried out with a half-normal probability plot (Abrahams & Keve, 1971) with all intramolecular distances less than 4·1 Å. The plot is linear with a correlation coefficient = 0·982, slope = 2·67 and intercept = –0·22. The interexperimental *R* value is 0·006. These results justify taking the average values to describe the molecular geometry. Tables 2 and 3 show the weighted mean values for bond lengths, bond angles and torsion angles. The corresponding weighted mean e.s.d.'s are corrected by the slope factor 2·27 (which indicates their underestimation).

The conformational analysis of the molecules gives the following results. The junction *A*/*B* in the decalin is *e–e trans*, with the sum of the internal torsion angles 114·4°. This value is slightly greater than that of 113·1° (Geise, Altona & Romers, 1966) for some *e–e trans*-decalins with the same degree of substitution at the bridgehead atoms. This could be due to the presence of the lactone bridge between C(4) and C(6). The conformations of rings *A* and *B* are described by Cremer & Pople's (1975) parameters. Ring *A* is a chair slightly distorted towards an envelope, defined by $\theta = 18°$, $\varphi = 309°$ [origin at C(1)] and $Q = 0{\cdot}59$ Å, which could also be due to the lactone bridge. Ring *B* with $\theta = 4°$, $\varphi = 1°$ [origin at C(9)] and $Q = 0{\cdot}51$ Å is a chair; the repulsion between O(8) and H(19) could be related to the ring flattening. Both γ-lactone rings have the envelope conformation with flaps at C(11) and C(5) respectively. The furan ring is planar. The acetyl group shows the usual *cis* C—O—C=O geometry. Both molecules of the asymmetric unit are linked together by two hydrogen bonds, O(1)*B*···O(7)*A* 3·029 (5), O(1)*A*···O(3)*B* 2·776 (4) Å.

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

$$\langle p\rangle = (p_1\sigma_1^{-2} + p_2\sigma_2^{-2})/(\sigma_1^{-2} + \sigma_2^{-2}).$$
$$\langle\sigma\rangle = 2{\cdot}67/(\sigma_1^{-2} + \sigma_2^{-2})^{1/2}.$$

O(1)–C(4)	1·441 (8)	C(4)–C(18)	1·530 (11)
O(2)–C(6)	1·475 (8)	C(5)–C(6)	1·524 (11)
O(2)–C(18)	1·349 (11)	C(5)–C(10)	1·565 (11)
O(3)–C(18)	1·208 (11)	C(5)–C(19)	1·546 (11)
O(4)–C(12)	1·464 (11)	C(6)–C(7)	1·513 (11)
O(4)–C(20)	1·359 (11)	C(7)–C(8)	1·524 (11)
O(5)–C(15)	1·350 (16)	C(8)–C(9)	1·552 (11)
O(5)–C(16)	1·372 (13)	C(8)–C(17)	1·537 (11)
O(6)–C(19)	1·446 (11)	C(9)–C(10)	1·564 (11)
O(6)–C(21)	1·348 (11)	C(9)–C(11)	1·545 (11)
O(7)–C(21)	1·199 (11)	C(9)–C(20)	1·528 (11)
O(8)–C(20)	1·198 (11)	C(11)–C(12)	1·532 (11)
C(1)–C(2)	1·542 (11)	C(12)–C(13)	1·479 (11)
C(1)–C(10)	1·539 (11)	C(13)–C(14)	1·444 (13)
C(2)–C(3)	1·507 (13)	C(13)–C(16)	1·337 (13)
C(3)–C(4)	1·522 (11)	C(14)–C(15)	1·332 (16)
C(4)–C(5)	1·536 (11)	C(21)–C(22)	1·494 (13)

C(6)–O(2)–C(18)	110·0 (5)	C(8)–C(9)–C(10)	108·2 (5)
C(12)–O(4)–C(20)	112·1 (5)	C(11)–C(9)–C(20)	102·7 (5)
C(15)–O(5)–C(16)	106·8 (11)	C(10)–C(9)–C(20)	114·6 (5)
C(19)–O(6)–C(21)	118·0 (5)	C(10)–C(9)–C(11)	109·7 (5)
C(2)–C(1)–C(10)	106·8 (5)	C(5)–C(10)–C(9)	117·5 (5)
C(1)–C(2)–C(3)	112·4 (8)	C(1)–C(10)–C(9)	117·2 (5)
C(2)–C(3)–C(4)	115·7 (8)	C(1)–C(10)–C(5)	110·5 (5)
O(1)–C(4)–C(3)	110·5 (5)	C(9)–C(11)–C(12)	105·7 (5)
C(3)–C(4)–C(18)	116·7 (8)	O(4)–C(12)–C(11)	104·6 (5)
C(3)–C(4)–C(5)	115·5 (5)	C(11)–C(12)–C(13)	115·0 (8)
O(1)–C(4)–C(18)	104·2 (5)	O(4)–C(12)–C(13)	108·6 (8)
O(1)–C(4)–C(5)	108·4 (5)	C(12)–C(13)–C(16)	126·6 (8)
C(5)–C(4)–C(18)	100·5 (5)	C(12)–C(13)–C(14)	127·6 (8)
C(4)–C(5)–C(19)	113·1 (5)	C(14)–C(13)–C(16)	105·7 (8)
C(4)–C(5)–C(10)	105·5 (5)	C(13)–C(14)–C(15)	106·6 (11)
C(4)–C(5)–C(6)	100·8 (5)	O(5)–C(15)–C(14)	110·6 (11)
C(10)–C(5)–C(19)	113·0 (5)	O(5)–C(16)–C(13)	110·3 (11)
C(6)–C(5)–C(19)	114·3 (5)	O(3)–C(18)–C(4)	128·8 (8)
C(6)–C(5)–C(10)	109·1 (5)	O(2)–C(18)–C(4)	109·2 (5)
O(2)–C(6)–C(5)	102·7 (5)	O(2)–C(18)–O(3)	121·9 (8)
C(5)–C(6)–C(7)	115·1 (5)	O(6)–C(19)–C(5)	109·7 (5)
O(2)–C(6)–C(7)	109·6 (5)	O(8)–C(20)–C(9)	129·6 (8)
C(6)–C(7)–C(8)	115·2 (5)	O(4)–C(20)–C(9)	110·7 (5)
C(7)–C(8)–C(17)	109·3 (8)	O(4)–C(20)–O(8)	119·6 (8)
C(7)–C(8)–C(9)	113·3 (5)	O(6)–C(21)–O(7)	124·0 (8)
C(9)–C(8)–C(17)	113·4 (5)	O(7)–C(21)–C(22)	125·9 (8)
C(8)–C(9)–C(20)	112·0 (5)	O(6)–C(21)–C(22)	110·0 (8)
C(8)–C(9)–C(11)	109·5 (5)		

Table 3. *Some average torsional angles* (°) *with e.s.d.'s in parentheses*

$\langle p\rangle$ and $\langle\sigma\rangle$ are as in Table 2.

C(10)–C(1)–C(2)–C(3)	–55·5 (9)	C(20)–C(9)–C(11)–C(12)	17·8 (7)
C(1)–C(2)–C(3)–C(4)	44·8 (10)	C(11)–C(12)–C(13)–C(14)	57·7 (12)
C(2)–C(3)–C(4)–C(5)	–43·6 (10)	C(12)–C(13)–C(14)–C(15)	–178·0 (9)
C(3)–C(4)–C(5)–C(10)	50·8 (8)	C(13)–C(14)–C(15)–O(5)	–0·3 (13)
C(4)–C(5)–C(10)–C(1)	–64·1 (7)	C(14)–C(15)–O(5)–C(16)	0·2 (13)
C(5)–C(10)–C(1)–C(2)	67·5 (8)	C(15)–O(5)–C(16)–C(13)	–0·0 (13)
C(1)–C(10)–C(5)–C(6)	–171·8 (6)	O(5)–C(16)–C(13)–C(14)	–0·2 (12)
C(9)–C(10)–C(5)–C(4)	157·8 (6)	C(16)–C(13)–C(14)–C(15)	0·3 (12)
C(10)–C(5)–C(6)–C(7)	–46·9 (8)	C(10)–C(9)–C(8)–C(17)	175·0 (6)
C(5)–C(6)–C(7)–C(8)	50·1 (9)	C(2)–C(3)–C(4)–O(1)	–167·2 (7)
C(6)–C(7)–C(8)–C(9)	–51·4 (9)	C(18)–C(4)–C(5)–C(6)	37·8 (7)
C(7)–C(8)–C(9)–C(10)	49·7 (8)	C(4)–C(5)–C(6)–O(2)	–38·5 (7)
C(8)–C(9)–C(10)–C(5)	–51·7 (8)	C(5)–C(6)–O(2)–C(18)	24·5 (7)
C(9)–C(10)–C(5)–C(6)	50·2 (8)	C(6)–O(2)–C(18)–C(4)	0·5 (8)
C(10)–C(9)–C(11)–C(12)	–104·5 (7)	O(2)–C(18)–C(4)–C(5)	–24·9 (8)
C(9)–C(11)–C(12)–O(4)	–20·3 (8)	C(1)–C(10)–C(5)–C(19)	59·9 (8)
C(11)–C(12)–O(4)–C(20)	15·3 (8)	C(10)–C(5)–C(19)–O(6)	33·1 (8)
C(12)–O(4)–C(20)–C(9)	–3·7 (9)	C(5)–C(19)–O(6)–C(21)	116·6 (7)
O(4)–C(20)–C(9)–C(11)	–9·2 (8)	C(19)–O(6)–C(21)–O(7)	7·9 (13)

The authors thank Professor S. García-Blanco for his support and the Centro de Procesado de Datos de JEN for computer facilities.

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Acta Cryst. (1981). B**37**, 2106–2108

The Structure of 2-Ammonio-γ-butyrolactone Bromide*

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(*Received* 10 *October* 1980; *accepted* 12 *May* 1981)

Abstract. $C_4H_8NO_2^+.Br^-$, M_r = 182·02, orthorhombic, *Pbca*, a = 11·786 (3), b = 9·499 (2), c = 11·594 (3) Å, Z = 8, D_c = 1·85 Mg m^{-3}, V = 1298·01 Å^3, λ(Cu $K\alpha$) = 1·5418 Å, μ = 8·058 mm^{-1}. The structure was solved by direct methods and refined by full-matrix least squares to a final R of 0·065 for 1024 observed reflections. The ring shows a typical envelope conformation while the lactone moiety is planar. The ammonio group, which in the solid state is bound to the bromine through hydrogen bonds, occupies a pseudo-axial position.

Introduction. The present study is part of a series of X-ray and NMR investigations on six- and five-membered rings carried out in our laboratories to compare the results obtained by these two different types of analysis and to have more data to check the Lambert theory (Lambert, 1967; Lambert & Sun, 1977).

A pale-yellow crystal of about 0·71 × 0·45 × 0·23 mm was selected, after recrystallization from a cyclohexane–diethyl ether mixture, to be used in the data collection.

Approximate cell dimensions were deduced from rotation and Weissenberg photographs. The cell constants were refined by a least-squares fit to the $(\theta,\chi,\varphi)_{hkl}$ values of 23 reflections carefully measured on the diffractometer.

During the data collection a check reflection was monitored periodically for crystal and instrument stability and did not show a significant variation. Using Cu $K\alpha$ radiation (λ = 1·5418 Å) a total of 2403 ($\pm h,k,l$) intensities with $\theta \leq 70°$ were measured on a Siemens AED single-crystal diffractometer. These were merged on 1235 unique reflections and 191 of them were considered unobserved on the basis that the intensities were less than $3\sigma(I)$.

The structure was solved by direct methods using *SHELX* (Sheldrick, 1975). Refinement of all heavy atoms began with a residual R of 0·22. A few cycles of isotropic full-matrix least squares reduced R to 0·99 and further cycles of anisotropic full-matrix refinement gave an R of 0·071. All the H atoms were located in the difference Fourier map computed at this stage. The final refinement including the H atoms with isotropic temperature factors reduced R and R_w to final values of 0·065 and 0·077 respectively. The weighting scheme used was $w = \sigma^2(F_o) + c|F_o|^2$, where c = 0·005. 20 reflections affected by large errors were removed from the final calculations.

Positional parameters are listed in Table 1.† All the calculations were performed on the Cyber 76 com-

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* Stereochemistry of Rings. IV. γ-Lactone Derivatives. 2. Part III.1: Bocelli & Grenier-Loustalot (1981). Part IV.1: Bocelli & Grenier-Loustalot (1980). Alternative name for title compound: 2-oxo-3-tetrahydrofurylammonium bromide.

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36104 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0567-7408/81/112106-03$01.00