tion, thermal motion and our atom-numbering scheme, and Fig. 3 displays the molecular packing. All computer programs from the *TEXSAN* crystal-structure-analysis package (Molecular Structure Corporation, 1985).

Related literature. In a similar compound (5a,11a-epoxy-5a,6a,7,10,10a,11a-hexahydro-7-methoxy-5,6,-9,11,12-naphthacenepentone), the same *cis* stereochemistry exists between the O atoms of the epoxide ring at C5a and C11a, and the H atoms at C6a and C10a (Gupta, Jackson & Stoodley, 1985).

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Structures of Three Pseudoguaianolides: Parthenin, Hymenolin (11β,13-Dihydroparthenin) and Bipinnatin

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Abstract. Parthenin, $C_{15}H_{18}O_4$, $M_r = 262{\cdot}3$, tetragonal, $P4_1$, $a = 6{\cdot}862\,(1)$, $c = 28{\cdot}681\,(8)$ Å, $V = 1350{\cdot}5\,(8)$ Å^3, $Z = 4$, $D_x = 1{\cdot}290$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}54184$ Å, $\mu = 7{\cdot}25$ cm^{-1}, $F(000) = 560$, $T = 298$ K, $R = 0{\cdot}036$ for 1316 observations with $I > 1\sigma(I)$. Hymenolin (11β,13-dihydroparthenin), $C_{15}H_{20}O_4$, $M_r = 264{\cdot}3$, monoclinic, $P2_1$, $a = 6{\cdot}495\,(1)$, $b = 28{\cdot}380\,(6)$, $c = 7{\cdot}587\,(1)$ Å, $\beta = 102{\cdot}90\,(1)°$, $V = 1363{\cdot}2\,(7)$ Å^3, $Z = 4$, $D_x = 1{\cdot}288$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}86$ cm^{-1}, $F(000) = 568$, $T = 297$ K, $R = 0{\cdot}060$ for 1757 observations with $I > 0{\cdot}5\sigma(I)$. Bipinnatin, $C_{15}H_{20}O_4$, $M_r = 264{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}231\,(1)$, $b = 9{\cdot}713\,(2)$, $c = 15{\cdot}453\,(2)$ Å, $V = 1385{\cdot}5\,(6)$ Å^3, $Z = 4$, $D_x = 1{\cdot}267$ g cm^{-3}, $\mu(\text{Cu } K\alpha) = 7{\cdot}07$ cm^{-1}, $F(000) = 568$, $T = 299$ K, $R = 0{\cdot}058$ for 1145 observations with $I > 3\sigma(I)$. The seven-membered ring of parthenin has a chair conformation with a pseudo mirror passing through C10, and asymmetry parameter $\Delta C_s = 4{\cdot}3°$. The cyclopentenone and lactone rings both have envelope conformations with C5 at the flap, $\Delta C_s = 5{\cdot}2°$ and C7 at the flap, $\Delta C_s = 0{\cdot}7°$, respectively. Molecules are linked in chains along the symmetry axis by hydrogen bonds involving the hydroxyl group and lactone carbonyl, O···O distance 2·805 (5) Å. The crystal structure of hymenolin contains two independent molecules. In both, the five-membered rings are in conformations closely resembling those of parthenin. The seven-membered ring of one molecule has a twist-chair conformation with C10 lying on the pseudo diad, and asymmetry parameter $\Delta C_2 = 3{\cdot}9°$, while the other molecule has an asymmetric seven-membered ring. Molecules are linked in chains of alternating molecule types, by hydrogen bonds involving hydroxy groups and lactone carbonyl O atoms. O···O distances are 2·855 (5) and 2·878 (5) Å. Hymenolin was isolated from *Hymenoclea salsola* T. and G. with unresolved stereochemistry at C11 [Toribio & Geissman (1968). *Phytochemistry*, **7**, 1623–1630]. The seven-membered ring of bipinnatin also has two conformations in the crystal, but both exist at the same site as a *ca* 70–30% disorder of atoms C8 and C9. The seven-membered ring of the major conformer has the parthenin conformation, with $\Delta C_s = 6{\cdot}1°$, while the minor conformer has the twist-chair

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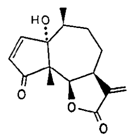
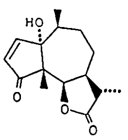
conformation of hymenolin, with $\Delta C_2 = 3{\cdot}1°$. The cyclopentanone ring has the envelope conformation with C2 at the flap and $\Delta C_s = 1{\cdot}3°$, while the lactone ring has the half-chair conformation with C12 on the pseudo diad, and $\Delta C_2 = 2{\cdot}7°$. Molecules are linked in chains along the **b** direction by hydrogen bonds involving the hydroxy group and the lactone carbonyl, with O···O distance 2·774 (7) Å.

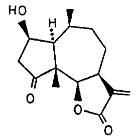
Experimental. Parthenin (I): crystal size $0{\cdot}28 \times 0{\cdot}40 \times 0{\cdot}52$ mm, space group determination by Laue symmetery $4/m$, systematic absence $00l$ with $l \neq 4n$ and presumption of absolute configuration as determined for bromoambrosin (Emerson, Herz, Caughlan & Witters, 1966), cell dimensions for setting angles of 25 reflections $70 > 2\theta > 66°$, data collection on Enraf–Nonius CAD-4 diffractometer, graphite monochromator, Cu$K\alpha$ radiation, ω–2θ scans designed to yield $I = 50\sigma(I)$, scan rates 0·20–5·0° min^{-1}. Data having $4 < 2\theta < 153°$, $0 \le h \le 8$, $0 \le k \le 8$, $0 \le l \le 36$ measured, corrected for background, Lorentz, polarization and absorption (ψ scans, minimum relative transmission 83·7%), 1456 unique data. Three standard reflections (200, 020, 008), no significant variation. Structure solved using *RANTAN* (Yao Jia-Xing, 1981), refined by full-matrix least squares based on F, $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$ with Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), data with $I > \sigma(I)$. Non-H atoms anisotropic; H atoms from difference maps, fixed contributions, $B = 5{\cdot}0$ Å^2. Secondary-extinction coefficient $4{\cdot}4\,(3) \times 10^{-6}$. Final $R = 0{\cdot}036$, $wR = 0{\cdot}037$, $s = 1{\cdot}230$ for 172 variables. Max. shift $0{\cdot}01\sigma$ in final cycle, max. residual density 0·10, min. $-0{\cdot}13$ e Å^{-3}.



(I)



(II)



(III)

Table 1. *Coordinates and equivalent isotropic thermal parameters for parthenin*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_i^*\mathbf{a}_i.\mathbf{a}_j.$
This expression for B_{eq} is also used in Tables 2 and 3.

	x	y	z	B_{eq}(Å^2)
O1	0·7510 (3)	0·3622 (3)	0	4·28 (6)
O2	0·9773 (3)	0·3323 (4)	0·0550 (8)	5·28 (7)
O3	0·4633 (4)	0·6423 (4)	−0·0305 (9)	7·85 (9)
O4	0·4712 (3)	0·2382 (3)	−0·1187 (7)	4·34 (6)
C1	0·6174 (4)	0·3827 (4)	−0·1294 (9)	3·49 (7)
C2	0·5084 (5)	0·5597 (5)	−0·1481 (12)	4·9 (1)
C3	0·4459 (5)	0·6734 (5)	−0·1137 (13)	5·8 (1)
C4	0·5179 (5)	0·5981 (4)	−0·0686 (12)	4·84 (9)
C5	0·6854 (4)	0·4533 (4)	−0·0804 (10)	3·26 (7)
C6	0·7021 (4)	0·2846 (4)	−0·0461 (9)	3·19 (7)
C7	0·8570 (4)	0·1238 (4)	−0·0561 (9)	3·26 (7)
C8	0·9681 (4)	0·1164 (4)	−0·1025 (10)	3·66 (7)
C9	0·8478 (5)	0·1020 (5)	−0·1470 (10)	4·13 (8)
C10	0·7596 (4)	0·2932 (4)	−0·1645 (10)	3·78 (8)
C11	0·9947 (5)	0·1449 (5)	−0·0160 (10)	4·04 (8)
C12	0·9151 (4)	0·2864 (4)	0·0171 (10)	3·84 (8)
C13	1·1655 (6)	0·0625 (7)	−0·0083 (14)	7·7 (1)
C14	0·9136 (5)	0·4329 (5)	−0·1839 (12)	5·1 (1)
C15	0·8703 (5)	0·5811 (4)	−0·0794 (12)	4·43 (9)

Table 2. *Coordinates and equivalent isotropic thermal parameters for hymenolin*

	x	y	z	B_{eq}(Å^2)
O1	0·8371 (6)	0	0·7039 (5)	4·04 (9)
O2	0·7966 (6)	−0·0597 (1)	0·5109 (5)	4·6 (1)
O3	0·9720 (8)	0·0342 (2)	1·0972 (6)	6·2 (1)
O4	0·6294 (5)	0·1281 (1)	0·8363 (5)	4·42 (9)
C1	0·8447 (8)	0·1314 (2)	0·8112 (7)	3·5 (1)
C2	0·977 (1)	0·1503 (2)	0·9861 (8)	4·6 (1)
C3	1·047 (1)	0·1182 (2)	1·1051 (8)	5·0 (2)
C4	0·9803 (9)	0·0720 (2)	1·0226 (7)	4·1 (1)
C5	0·9154 (8)	0·0801 (2)	0·8156 (7)	3·4 (1)
C6	0·7425 (8)	0·0458 (2)	0·7245 (7)	3·4 (1)
C7	0·6082 (8)	0·0582 (2)	0·5337 (7)	3·3 (1)
C8	0·7009 (9)	0·0936 (2)	0·4196 (8)	4·3 (2)
C9	0·686 (1)	0·1453 (2)	0·4760 (9)	5·1 (2)
C10	0·8381 (9)	0·1631 (2)	0·6454 (8)	4·4 (1)
C11	0·5711 (9)	0·0096 (2)	0·4439 (7)	3·7 (1)
C12	0·7436 (8)	−0·0210 (2)	0·5493 (7)	3·4 (1)
C13	0·357 (1)	−0·0120 (2)	0·4470 (8)	4·7 (2)
C14	1·054 (1)	0·1758 (3)	0·6090 (9)	5·8 (2)
C15	1·1276 (9)	0·0711 (2)	0·7569 (8)	4·7 (2)
O1′	0·2781 (6)	0·7516 (1)	−0·0282 (5)	4·29 (9)
O2′	0·5413 (6)	0·7020 (2)	−0·0151 (6)	5·5 (1)
O3′	0·0047 (7)	0·7916 (2)	0·2006 (6)	6·5 (1)
O4′	−0·0340 (6)	0·8701 (1)	−0·2224 (5)	3·94 (9)
C1′	0·1362 (8)	0·8823 (2)	−0·0718 (7)	3·2 (1)
C2′	0·0312 (8)	0·9072 (2)	0·0651 (8)	3·9 (1)
C3′	−0·0149 (8)	0·8756 (2)	0·1812 (8)	4·2 (1)
C4′	0·0568 (8)	0·8282 (2)	0·1473 (8)	4·3 (1)
C5′	0·2145 (8)	0·8359 (2)	0·0218 (7)	3·1 (1)
C6′	0·1979 (8)	0·7952 (2)	−0·1177 (7)	3·3 (1)
C7′	0·3194 (8)	0·7993 (2)	−0·2726 (7)	3·9 (1)
C8′	0·345 (1)	0·8475 (2)	−0·3583 (7)	4·6 (2)
C9′	0·4421 (9)	0·8865 (2)	−0·2341 (7)	3·7 (1)
C10′	0·2936 (8)	0·9144 (2)	−0·1407 (7)	3·5 (1)
C11′	0·5254 (8)	0·7746 (2)	−0·1948 (7)	3·6 (1)
C12′	0·4584 (9)	0·7374 (2)	−0·0727 (8)	4·0 (1)
C13′	0·635 (1)	0·7516 (2)	−0·3329 (9)	5·0 (2)
C14′	0·4221 (9)	0·9483 (2)	−0·0003 (9)	4·8 (2)
C15′	0·4307 (8)	0·8379 (2)	0·1559 (7)	3·4 (1)

Hymenolin (11β,13-dihydroparthenin) (II): crystal size $0{\cdot}20 \times 0{\cdot}40 \times 0{\cdot}56$ mm, space group from Laue symmetry $2/m$, systematic absences $0k0$ with k odd and known chirality of material, $20 \le 2\theta \le 24°$ for cell dimensions, data collection as for parthenin, Mo $K\alpha$ radiation, scan rates 0·49–5·0° min^{-1}, $2 < 2\theta < 50°$, $0 \le h \le 7$, $0 \le k \le 33$, $-9 \le l \le 9$, no absorption correction, 2440 unique data ($R_{int} = 0{\cdot}025$). Standards (200, 040, 001), no decay. Solved using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refined with $I > 0{\cdot}5\sigma(I)$ data, non-H atoms anisotropic, H atoms located by ΔF synthesis, fixed contributions, $B = 5{\cdot}0$ Å^2, $R = 0{\cdot}060$, $wR = 0{\cdot}054$, $S = 1{\cdot}812$, 342 variables. Max. shift $< 0{\cdot}01\sigma$, max. residual density 0·22, min. $-0{\cdot}26$ e Å^{-3}.

Bipinnatin (III): crystal size $0{\cdot}21 \times 0{\cdot}38 \times 0{\cdot}56$ mm, absences $h00$ with h odd, $0k0$ with k odd, $00l$ with l odd, $60 < 2\theta < 67°$ for cell dimensions, data collection as for parthenin, Cu $K\alpha$ radiation, scan rates 0·71–4·0° min^{-1}, $4 < 2\theta < 150°$, $0 \le h \le$

Table 3. *Coordinates and equivalent isotropic thermal parameters for bipinnatin*

	x	y	z	B_{eq}(Å^2)
O1	0·1502 (4)	0·7394 (4)	0·4912 (2)	5·81 (8)
O2	0·0585 (4)	0·9433 (4)	0·4595 (3)	7·7 (1)
O3	0·0028 (4)	0·4974 (4)	0·5642 (2)	7·1 (1)
O4	0·0673 (4)	0·2136 (4)	0·4019 (3)	6·38 (9)
C1	0·2565 (5)	0·3891 (5)	0·4033 (3)	4·5 (1)
C2	0·1974 (6)	0·2576 (5)	0·4439 (4)	5·8 (1)
C3	0·1607 (6)	0·3064 (6)	0·5350 (3)	6·2 (1)
C4	0·0944 (4)	0·4447 (5)	0·5197 (3)	5·2 (1)
C5	0·1524 (5)	0·5058 (5)	0·4331 (3)	4·07 (9)
C6	0·2413 (5)	0·6328 (5)	0·4538 (3)	4·8 (1)
C7	0·3199 (6)	0·7089 (6)	0·3771 (4)	6·3 (1)
C10	0·3122 (6)	0·3805 (6)	0·3075 (3)	5·9 (1)
C11	0·2294 (6)	0·8301 (6)	0·3630 (4)	5·9 (1)
C12	0·1346 (6)	0·8493 (5)	0·4408 (4)	5·6 (1)
C13	0·2243 (7)	0·9182 (7)	0·2966 (4)	7·6 (2)
C14	0·2039 (8)	0·3368 (7)	0·2382 (4)	7·6 (2)
C15	0·0172 (5)	0·5357 (5)	0·3775 (3)	5·1 (1)
C8*A*	0·3373 (9)	0·6319 (8)	0·2830 (5)	5·8 (2)*
C8*B*	0·4229 (20)	0·6268 (19)	0·3243 (11)	6·1 (4)*
C9*A*	0·4120 (9)	0·4940 (9)	0·2892 (5)	6·1 (2)*
C9*B*	0·3461 (18)	0·5417 (17)	0·2692 (9)	4·7 (3)*

* Starred atoms were refined isotropically, and isotropic B is given. Atoms labelled with A have population 0·70, those with B 0·30.

11, $0 \leq k \leq 12$, $0 \leq l \leq 19$, minimum relative transmission 76·92%, 1652 unique data. Standards (200, 040, 006), no decay. Solved using *MULTAN*78 (Main *et al.*, 1978), refined with $I > 3\sigma(I)$ data, non-H atoms anisotropic except for disorder region, H atoms located in difference maps, fixed contributions $B = 7{\cdot}0$ Å^2. Atoms C8 and C9 disordered into two orientations, with ~70% occupancy (C8*A* and C9*A*) and ~30% (C8*B* and C9*B*); These refined isotropically. Final $R = 0{\cdot}058$, $wR = 0{\cdot}049$, $S = 1{\cdot}726$, 170 variables. Max. shift 0·23σ, max. residual density 0·20, min. $-0{\cdot}18$ e Å^{-3}.

Atomic parameters for non-H atoms are given in Tables 1–3; the molecules are depicted in Figs. 1–4. Conformations of molecules are specified by torsion

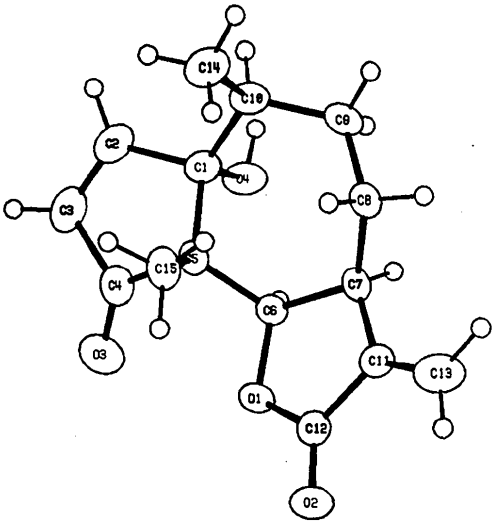


Fig. 1. *ORTEP* (Johnson, 1976) representation of parthenin.

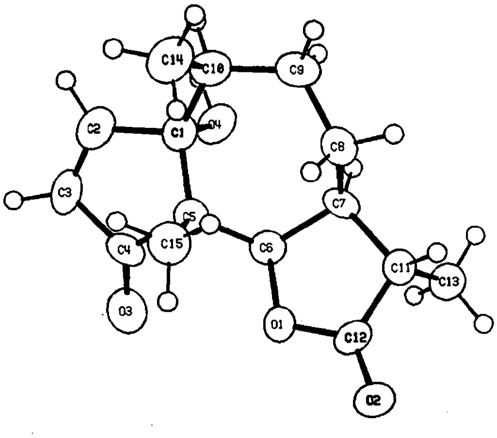


Fig. 2. *ORTEP* drawing of hymenolin (11β,13-dihydroparthenin), unprimed molecule.

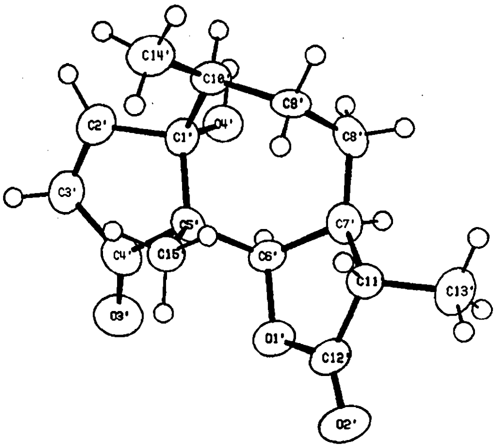


Fig. 3. *ORTEP* drawing of hymenolin (11β,13-dihydroparthenin), primed molecule.

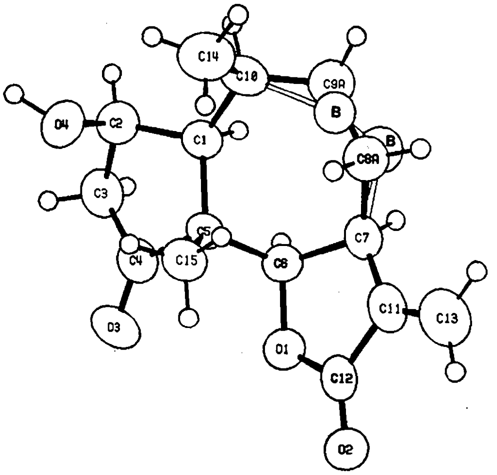


Fig. 4. *ORTEP* representation of bipinnatin. The minor (30%) conformer is labelled *B*.

Table 4. *Torsion angles* (°)

E.s.d.'s ~0·6° for parthenin, 0·6° for hymenolin and 0·8° for the ordered portion of bipinnatin. Torsion angles involving disordered atoms of bipinnatin have e.s.d.'s ~1° for those involving major contributor *A*, and ~3° for those involving minor contributor *B* (in parentheses).

	Parthenin	Hymenolin	Hymenolin′	Bipinnatin	
C1—C2—C3—C4	4·8	3·1	2·6	41·4	
C2—C3—C4—C5	16·1	16·2	15·3	−25·7	
C3—C4—C5—C1	−28·7	−28·3	−26·1	−0·4	
C4—C5—C1—C2	30·0	28·3	27·0	26·7	
C5—C1—C2—C3	−23·3	−21·5	−19·7	−43·0	
C1—C5—C6—C7	64·3	47·8	59·2	67·7	
C5—C6—C7—C8	−9·3	21·1	−35·0	−14·6	(−57·4)
C6—C7—C8—C9	−57·3	−77·5	54·8	−55·7	(75·8)
C7—C8—C9—C10	80·0	76·0	−83·8	87·0	(−92·9)
C8—C9—C10—C1	−61·6	−50·9	42·0	−71·5	(45·9)
C9—C10—C1—C5	63·6	61·9	42·5	69·3	(38·4)
C10—C1—C5—C6	−81·1	−82·7	−88·8	−82·8	
O1—C6—C7—C11	−6·8	21·9	−29·6	−18·4	
C6—C7—C11—C12	6·6	−21·3	31·1	16·5	
C7—C11—C12—O1	−4·0	13·9	−22·8	−8·4	
C11—C12—O1—C6	−0·8	0·7	3·7	−4·7	
C12—O1—C6—C7	4·9	−14·8	16·4	14·8	
O2—C12—C11—C13	−7·0	69·5	35·1	−8·3	

Table 5. *Bond distances* (Å)

In the disordered region of bipinnatin, only distances involving the major conformer (*A*) are given.

	Parthenin	Hymenolin	Hymenolin′	Bipinnatin
O1—C6	1·464 (5)	1·460 (5)	1·451 (5)	1·454 (7)
O1—C12	1·334 (5)	1·335 (5)	1·351 (6)	1·329 (8)
O2—C12	1·211 (6)	1·206 (5)	1·177 (6)	1·188 (8)
O3—C4	1·194 (7)	1·220 (6)	1·191 (6)	1·205 (8)
O4—C1	1·444 (5)	1·456 (5)	1·444 (5)	—
O4—C2	—	—	—	1·430 (8)
C1—C2	1·524 (7)	1·510 (7)	1·537 (6)	1·524 (9)
C1—C5	1·559 (6)	1·524 (7)	1·527 (6)	1·555 (8)
C1—C10	1·530 (6)	1·539 (7)	1·545 (6)	1·570 (8)
C2—C3	1·330 (8)	1·289 (8)	1·337 (7)	1·524 (9)
C3—C4	1·477 (7)	1·476 (7)	1·466 (7)	1·495 (9)
C4—C5	1·556 (7)	1·549 (7)	1·562 (7)	1·559 (8)
C5—C6	1·524 (6)	1·530 (6)	1·554 (6)	1·516 (8)
C5—C15	1·542 (6)	1·561 (6)	1·540 (6)	1·543 (8)
C6—C7	1·559 (6)	1·554 (6)	1·559 (7)	1·574 (9)
C7—C8	1·536 (6)	1·535 (7)	1·539 (7)	1·64 (2)
C7—C11	1·495 (7)	1·532 (7)	1·510 (6)	1·460 (10)
C8—C9	1·523 (7)	1·539 (8)	1·497 (7)	1·51 (2)
C9—C10	1·530 (7)	1·522 (7)	1·538 (6)	1·46 (2)
C10—C14	1·532 (7)	1·531 (7)	1·536 (7)	1·524 (10)
C11—C12	1·465 (6)	1·501 (7)	1·529 (7)	1·499 (9)
C11—C13	1·320 (7)	1·526 (7)	1·538 (7)	1·338 (9)

angles in Table 4, while bond distances and angles are listed in Tables 5–7.*

Related literature. Various plant sources for parthenin (Fischer, Olivier & Fischer, 1979); characterization of parthenin (Herz, Watanabe, Miyazaki & Kishida, 1962); isolation of hymenolin from *Hymenoclea salsola,* and characterization (Toribio & Geissman, 1968; Balza & Towers, 1988); isolation of bipinnatin from *Parthenium bipinnatifidum,* and characterization (Rodriguez, Yoshioka & Mabry, 1971); crystal structure of stramonin-B (Fortier, DeTitta & Grieco, 1979); crystal structure of hysterin

Table 6. *Bond angles* (°) *in parthenin and hymenolin*

	Parthenin	Hymenolin	Hymenolin′
C6—O1—C12	112·6 (4)	111·8 (4)	112·2 (4)
O4—C1—C2	106·4 (4)	106·5 (4)	105·6 (4)
O4—C1—C5	103·3 (4)	103·3 (4)	106·1 (4)
O4—C1—C10	107·9 (4)	107·0 (4)	108·7 (4)
C2—C1—C5	102·5 (4)	102·1 (4)	103·6 (4)
C2—C1—C10	113·7 (4)	114·7 (5)	112·6 (4)
C5—C1—C10	121·8 (4)	112·0 (4)	119·3 (4)
C1—C2—C3	111·3 (5)	114·1 (5)	109·6 (5)
C2—C3—C4	109·7 (5)	107·8 (5)	111·9 (5)
O3—C4—C3	127·4 (5)	128·6 (5)	128·0 (5)
O3—C4—C5	126·3 (5)	124·8 (5)	127·1 (5)
C3—C4—C5	106·3 (5)	106·6 (5)	104·8 (5)
C1—C5—C4	100·0 (4)	100·1 (4)	102·0 (4)
C1—C5—C6	111·7 (4)	114·2 (4)	111·0 (4)
C1—C5—C15	116·1 (4)	115·5 (4)	115·9 (4)
C4—C5—C6	113·6 (4)	111·7 (4)	110·8 (4)
C4—C5—C15	103·9 (4)	102·0 (4)	103·2 (4)
C6—C5—C15	111·0 (4)	111·9 (4)	113·0 (4)
O1—C6—C5	109·0 (4)	109·3 (4)	110·6 (4)
O1—C6—C7	105·5 (4)	105·3 (4)	103·1 (4)
C5—C6—C7	118·0 (4)	118·6 (4)	119·2 (4)
C6—C7—C8	121·4 (4)	117·7 (4)	120·5 (4)
C6—C7—C11	102·8 (4)	102·1 (4)	103·0 (4)
C8—C7—C11	110·9 (4)	112·3 (4)	114·0 (4)
C7—C8—C9	117·4 (4)	114·2 (4)	117·4 (4)
C8—C9—C10	115·7 (4)	118·7 (5)	116·9 (4)
C1—C10—C9	112·4 (4)	113·0 (4)	112·5 (4)
C1—C10—C14	115·3 (4)	115·2 (4)	115·7 (4)
C9—C10—C14	112·5 (5)	112·0 (5)	110·0 (4)
C7—C11—C12	109·1 (4)	105·0 (4)	102·2 (4)
C7—C11—C13	130·4 (5)	114·2 (4)	115·9 (4)
C12—C11—C13	120·5 (5)	109·5 (4)	111·1 (4)
O1—C12—O2	121·8 (5)	121·4 (5)	120·8 (5)
O1—C12—C11	109·5 (4)	110·4 (4)	109·0 (5)
O2—C12—C11	128·6 (5)	128·2 (5)	130·2 (5)

Table 7. *Bond angles* (°) *in bipinnatin*

C6—O1—C12	113·7 (5)	C6—C7—C11	103·1 (6)
C2—C1—C5	105·6 (5)	C6—C7—C8*A*	119·8 (6)
C2—C1—C10	117·4 (6)	C6—C7—C8*B*	117·2 (11)
C5—C1—C10	121·3 (5)	C11—C7—C8*A*	106·9 (7)
O4—C2—C1	111·4 (5)	C11—C7—C8*B*	135·8 (12)
O4—C2—C3	109·0 (6)	C1—C10—C14	117·6 (6)
C1—C2—C3	101·5 (6)	C1—C10—C9*A*	110·4 (7)
C2—C3—C4	102·9 (6)	C1—C10—C9*B*	109·8 (9)
O3—C4—C3	125·3 (7)	C14—C10—C9*A*	119·1 (7)
O3—C4—C5	124·8 (6)	C14—C10—C9*B*	97·6 (9)
C3—C4—C5	109·7 (6)	C7—C11—C12	108·4 (7)
C1—C5—C4	100·9 (5)	C7—C11—C13	130·6 (7)
C1—C5—C6	108·7 (5)	C12—C11—C13	121·0 (7)
C1—C5—C15	118·2 (5)	O1—C12—O2	122·6 (7)
C4—C5—C6	108·3 (5)	O1—C12—C11	107·9 (6)
C4—C5—C15	105·7 (5)	O2—C12—C11	129·5 (7)
C6—C5—C15	113·7 (5)	C7—C8*A*—C9*A*	113·1 (8)
O1—C6—C5	110·5 (5)	C7—C8*B*—C9*B*	109 (2)
O1—C6—C7	103·4 (5)	C10—C9*A*—C8*A*	113·1 (8)
C5—C6—C7	118·2 (5)	C10—C9*B*—C8*B*	116 (2)

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* Tables of H-atom coordinates, anisotropic thermal parameters and structure factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52087 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Declercq, Germain, Van Meerssche, Demuynck, De Clercq & Vandewalle, 1980); crystal structure of psilostachyin-A (Wilzer, Han, Zambrano, Fronczek & Watkins, 1988); crystal structure of confertiflorin (Vargas, Fronczek, Fischer & Hostettmann, 1986); conformational asymmetry parameters (Duax & Norton, 1975).

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1,4-Dibromohomocubane Ethylene Ketal

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Abstract. 1,4-Dibromopentacyclo[4.3.0.$0^{2,5}$.$0^{3,8}$.$0^{4,7}$]nonan-9-one ethylene ketal (1), $C_{11}H_{10}Br_2O_2$, M_r = 334·02, monoclinic, $P2_1/a$, a = 12·651 (4), b = 6·197 (1), c = 14·301 (3) Å, β = 107·15 (2)°, V = 1071·4 (4) Å^3, Z = 4, D_x = 2·071 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 74·7 cm^{-1}, F(000) = 648, T = 295 K, R = 0·0664 for 1365 reflections. The cage structure consists of four four-membered rings fused to a norbornane moiety (two fused five-membered rings) with an ethylene ketal attached to the methylene bridge of the norbornane. Two four-membered rings are planar and two are folded along a diagonal. The five-membered ethylene ketal ring is in an envelope conformation but the flap is not at the spiro fusion center with the cage. Molecular mechanics calculations give ΔH_f = 168·3 kJ mol^{-1} and E(strain) = 619·3 kJ mol^{-1} with major contributions from angle (359·8 kJ mol^{-1}) and torsional strain (272·0 kJ mol^{-1}).

Experimental. The title compound was synthesized by literature procedures (Chapman, Key & Toyne, 1970; Mehta, Srikrishna & Suri, 1980), and recrystallization yielded a colorless, poor-quality crystal (asymmetric peak profiles and backgrounds with some diffraction spots exceeding maximum scan width) of dimensions 0·50 × 0·43 × 0·35 mm. After data collection an attempt was made to cleave the crystal to a smaller size to reduce the absorption correction; however, the crystal shattered into small fragments. All data were collected on a Nicolet $R3M/\mu$ update of a $P2_1$ diffractometer, ω scan technique ($3 \leq 2\theta \leq 50°$), variable scan rate of 4 to 29·3° min^{-1}, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from a least-squares refinement of 25 reflections ($23{\cdot}16 \leq 2\theta \leq 28{\cdot}84°$);

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