values of the anisotropic temperature factors are given in Table 1; selected bond distances and angles are listed in Table 2.*

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond lengths and angles, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54416 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The structure determination is part of our studies on the synthesis of taxane diterpenoids (Horiguchi, Furukawa & Kuwajima, 1989).

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Micheliolide

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Abstract. $C_{15}H_{20}O_3$, $M_r = 248.3$, orthorhombic, $C222_1$, $a = 7.5919$ (7), $b = 15.5508$ (7), $c = 22.349$ (3) Å, $V = 2638.5$ (7) Å^3, $Z = 8$, $D_x = 1.250$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 0.65$ mm^{-1}, $F(000) = 1072$, $T = 294$ K, $R = 0.030$ for 1488 observations with $I > 3\sigma(I)$ (of 1544 unique data). The seven-membered ring is *trans* fused to the lactone ring. The two five-membered rings are in half-chair conformations while the seven-membered ring is in a distorted-chair conformation, with the pseudomirror bisecting the double bond. The C14 methyl group is disordered into two rotamers. Molecules form weakly hydrogen-bonded dimers about twofold axes, in which the OH H atom is disordered. The hydroxy group donates an intermolecular bifurcated hydrogen bond to both O atoms of the lactone ring [O···O (carbonyl) 3.399 (2), O···O (ring) 3.131 (2) Å] and accepts a second hydrogen bond from the hydroxy group of the same molecule [O···O 3.004 (2) Å].

Experimental. Micheliolide (1) is the major compound obtained from the BF_3-mediated rearrangement of parthenolide (Parodi, Fronczek & Fischer, 1989).

Crystals formed from ethyl acetate–hexane solution, m.p. 415–418 K, were suitable; a clear colorless crystal with dimensions 0.25 × 0.40 × 0.40 mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $30 > \theta > 25°$. The ω–2θ scans were designed for $I = 50\sigma(I)$, subject to max. scan time = 120 s, scan rates varied from 0.53–3.30° min^{-1}. An octant of data having $h + k$ even ($2 < \theta < 75°$) $0 \le h \le 9$, $0 \le k \le 19$, $0 \le l \le 28$ was measured and corrected for background, Lorentz, polarization and absorption. Absorption corrections were based on ψ scans, with min. relative transmission coefficient 96.46%. Three standard reflections (600, 0,10,0, 008) exhibited no significant variation in intensity, and no decay correction was applied. 1544 unique reflections were measured. Systematic absences hkl with $h + k$ odd and $00l$ with l odd indicated space group $C222_1$. The structure was solved by direct methods using *RANTAN* (Yao, 1981), refined by full-matrix least squares based upon F, using data for which $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using the *Enraf–Nonius Structure Determination Package* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), and anomalous coefficients of Cromer (1974). Heavy-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF synthesis and except as noted below were refined with isotropic thermal parameters. The hydroxy-H atom is disordered into two half-populated sites; both were refined isotropically. Methyl group C14 is also disordered into two rotamers. Six half-populated H atoms were included as fixed contributors. Final $R = 0.030$ for 1488 observed data (0.031 for all 1544 data), $wR = 0.043$ and $S = 3.141$ for 236 variables. Max. shift 0.03σ in the final cycle, max. residual density 0.13, min. -0.13 e Å^{-3}, and extinction coefficient $g = 3.2\ (2) \times 10^{-6}$ where the factor $(1 + gI_c)^{-1}$ was applied to F_c. The fractional coordinates of the title compound are given in Table 1. A structural diagram is given

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Table 1. *Coordinates and equivalent isotropic thermal parameters*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}(Å^2)
O1	0.2255 (2)	0.15392 (6)	0.01920 (4)	4.00 (2)
O2	0.2167 (2)	0.21934 (8)	−0.06945 (4)	5.55 (3)
O3	0.0864 (2)	−0.01026 (7)	0.06682 (4)	5.18 (2)
C1	0.2189 (2)	0.1305 (1)	0.18918 (5)	3.73 (2)
C2	0.2360 (3)	0.0419 (1)	0.21760 (6)	5.29 (4)
C3	0.1630 (3)	−0.0207 (1)	0.17168 (7)	5.14 (4)
C4	0.2067 (3)	0.01980 (9)	0.11135 (5)	4.03 (3)
C5	0.1700 (2)	0.11680 (9)	0.12337 (5)	3.27 (2)
C6	0.2598 (2)	0.17855 (8)	0.08159 (5)	3.25 (2)
C7	0.1963 (2)	0.27159 (9)	0.08544 (6)	3.53 (2)
C8	0.2810 (3)	0.32066 (9)	0.13597 (7)	4.44 (3)
C9	0.2123 (3)	0.2940 (1)	0.19726 (7)	5.43 (4)
C10	0.2384 (2)	0.2034 (1)	0.21973 (6)	4.20 (3)
C11	0.2289 (2)	0.30177 (9)	0.02282 (6)	3.88 (3)
C12	0.2231 (2)	0.2244 (1)	−0.01579 (6)	3.98 (3)
C13	0.2656 (3)	0.3793 (1)	0.00143 (9)	5.52 (4)
C14	0.2812 (3)	0.1995 (1)	0.28614 (6)	5.67 (4)
C15	0.3961 (3)	0.0018 (1)	0.09335 (7)	5.26 (4)

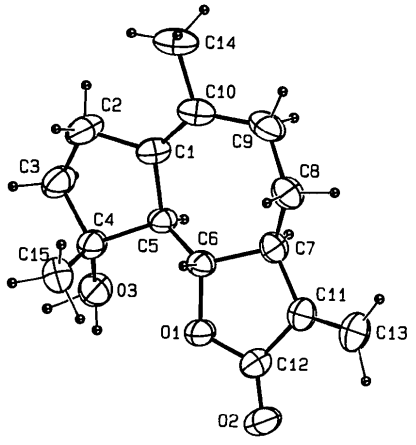


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule, representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radius. Both half-populated H atoms on O3 are illustrated, only one set of those on C14.

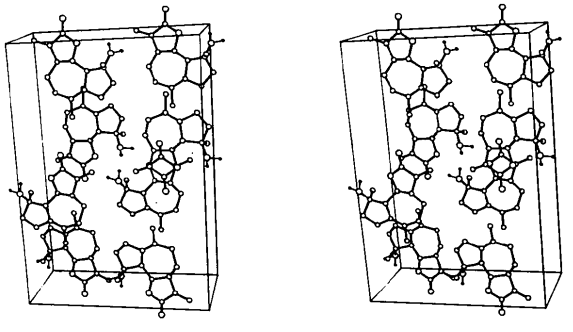


Fig. 2. Stereoview of the unit cell, viewed slightly oblique to the *a* axis. Only those H atoms involved in hydrogen bonding are illustrated.

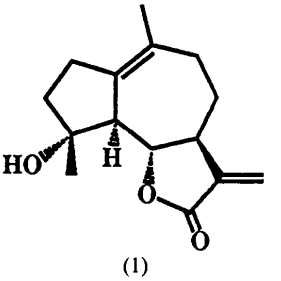
Table 2. *Bond distances* (Å), *angles* (°) *and selected torsion angles* (°)

O1—C6	1.469 (1)	C4—C15	1.519 (3)
O1—C12	1.347 (2)	C5—C6	1.503 (2)
O2—C12	1.203 (2)	C6—C7	1.528 (2)
O3—C4	1.429 (2)	C7—C8	1.507 (2)
C1—C2	1.522 (2)	C7—C11	1.497 (2)
C1—C5	1.532 (2)	C8—C9	1.523 (2)
C1—C10	1.332 (2)	C9—C10	1.509 (2)
C2—C3	1.520 (2)	C10—C14	1.521 (2)
C3—C4	1.525 (2)	C11—C12	1.481 (2)
C4—C5	1.557 (2)	C11—C13	1.327 (2)

C6—O1—C12	109.9 (1)	O1—C6—C7	104.1 (1)
C2—C1—C5	107.2 (1)	C5—C6—C7	115.3 (1)
C2—C1—C10	123.1 (1)	C6—C7—C8	112.8 (1)
C5—C1—C10	129.6 (1)	C6—C7—C11	101.1 (1)
C1—C2—C3	105.5 (1)	C8—C7—C11	118.1 (1)
C2—C3—C4	104.6 (1)	C7—C8—C9	113.0 (1)
O3—C4—C3	110.0 (1)	C8—C9—C10	120.6 (1)
O3—C4—C5	108.8 (1)	C1—C10—C9	127.6 (1)
O3—C4—C15	111.1 (1)	C1—C10—C14	119.4 (1)
C3—C4—C5	102.1 (1)	C9—C10—C14	112.9 (1)
C3—C4—C15	111.3 (1)	C7—C11—C12	106.6 (1)
C5—C4—C15	113.2 (1)	C7—C11—C13	131.0 (1)
C1—C5—C4	104.9 (1)	C12—C11—C13	122.3 (1)
C1—C5—C6	113.4 (1)	O1—C12—O2	121.7 (1)
C4—C5—C6	115.5 (1)	O1—C12—C11	108.8 (1)
O1—C6—C5	110.0 (1)	O2—C12—C11	129·5 (1)

C13—C11—C12—O2	−14.8 (3)	O1—C6—C7—C11	−30.1 (1)
C5—C6—C7—C8	82.2 (2)	C2—C1—C5—C4	−14.1 (2)
C5—C1—C10—C9	1.7 (3)	C10—C1—C5—C6	41.5 (2)

below. Fig. 1 is a perspective drawing showing the atom numbering and Fig. 2 illustrates the unit cell. Bond distances, angles and selected torsion angles are presented in Table 2.*



(1)

Related literature. Bond lengths C11—C13 1.327 (2), C12—O2 1.203 (2) Å of the title molecule are similar to those of 7α-hydroxy-3-desoxyzaluzanin C [C11—C13 1.323 (4), C12—O2 1.213 (4) Å] (Fronczek, Vargas & Fischer, 1984), 8β-angeloyloxymaximilianin [C11—C13 1.308 (4), C12—O2 1.207 (3) Å] (Watson & Zabel, 1982) and acroptilin [C11—C13: 1.321 (6) Å; C12—O2: 1.207 (6) Å] (Stevens & Wong, 1982). The lactone exocyclic torsion angle

* Lists of H-atom coordinates and thermal parameters, bond distances and angles involving H atoms, anisotropic thermal parameters, torsion angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54425 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

O2—C12—C11—C13 and the torsion angle C11—C7—C6—O1 at the lactone seven membered ring fusion bond are −14.8 (3) and −30.1 (1)°, respectively, and vary with those of 8β-angeloyloxymaximilianin [−2.8 (3) and −13.1 (2)° respectively] (Watson & Zabel, 1982), bahia I [−11.5 (6) and −18.5 (3)° respectively] (Herz, Govindan & Blount, 1980) and 7α-hydroxy-3-desoxyzaluzanin C [−19.5 (4), −30.8 (4), and −6.8 (4) and −24.2 (4)° respectively]. Two independent molecules in the unit cell (Fronczek, Vargas & Fischer, 1984).

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Structure of 1-[(4-Acetamidophenyl)thio]-3-[4-(3-methylphenyl)piperazin-1-yl]propane Monohydrate

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Abstract. $C_{22}H_{29}N_3OS.H_2O$, $M_r = 401.57$, monoclinic, $P2_1/c$, $a = 6.511$ (1), $b = 14.914$ (3), $c = 22.550$ (4) Å, $\beta = 97.84$ (1)°, $V = 2169.1$ Å^3, $Z = 4$, $D_x = 1.23$ g cm^{-3}, $\bar{\lambda}$(Cu $K\alpha$) = 1.54178 Å, $\mu = 14.59$ cm^{-1}, $F(000) = 864$, room temperature, $R = 0.058$ for 1270 observed reflections. This compound is a centrally active hypotensive agent. The molecule can be described by two planar moieties, *i.e.* the methylphenyl group and the [(4-acetamidophenyl)thio]propane group substituting a piperazine ring in a chair conformation. The overall conformation is *trans* extended.

Experimental. Colorless prism, dimensions 0.30 × 0.20 × 0.15 mm. Density not measured. Unit-cell parameters and intensity data obtained from an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Cu $K\alpha$ radiation in ω/θ scan mode ($0 < \theta < 65°$). Cell dimensions refined by least-squares fitting of θ values of 22 reflections. No appreciable drop in intensity of a standard reflection ($14\bar{1}$) checked every 3600 s. 3690 independent reflections collected in $\pm h, k, l$, range −11,0,0 to 11,11,27; 1270 observed reflections with $I > 3\sigma(I)$ used in subsequent calculations. Intensities corrected for Lorentz and polarization effects but not for absorption. Scattering factors for non-H atoms from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 201–209) and for H from Stewart, Davidson & Simpson (1965). Structure solved with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and standard Fourier synthesis techniques. H atoms located by ΔF synthesis and refined. Block-diagonal-matrix least-squares refinement on F of observed reflections, $w = 1$ if $F_o < P$, $P = [F_o^2(\text{max.})/10]^2$, $w = (P/F_0)^2$ if $F_o > P$;

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