

Table 3. *Interatomic distances (Å) and angles (°)*

P—O	1.482 (2)	P—C(1)	1.793 (2)
P—C(11)	1.812 (2)	P—C(21)	1.807 (2)
C(1)—C(2)	1.525 (3)	C(2)—C(2')	1.521 (3)
C(11)—C(12)	1.381 (4)	C(11)—C(16)	1.382 (4)
C(12)—C(13)	1.377 (4)	C(13)—C(14)	1.361 (5)
C(14)—C(15)	1.358 (5)	C(15)—C(16)	1.383 (4)
C(21)—C(22)	1.381 (4)	C(21)—C(26)	1.387 (3)
C(22)—C(23)	1.382 (4)	C(23)—C(24)	1.368 (4)
C(24)—C(25)	1.367 (5)	C(25)—C(26)	1.381 (4)
O—P—C(1)	114.9 (1)	O—P—C(11)	111.00 (9)
O—P—C(21)	112.45 (9)	C(1)—P—C(11)	105.6 (1)
C(1)—P—C(21)	106.9 (1)	C(11)—P—C(21)	105.3 (1)
P—C(1)—C(2)	112.0 (2)	C(1)—C(2)—C(2')	113.1 (2)
P—C(11)—C(12)	123.7 (2)	P—C(11)—C(16)	118.1 (2)
C(12)—C(11)—C(16)	118.3 (2)	C(11)—C(12)—C(13)	120.1 (2)
C(12)—C(13)—C(14)	121.1 (3)	C(13)—C(14)—C(15)	119.6 (3)
C(14)—C(15)—C(16)	120.2 (3)	C(11)—C(16)—C(15)	120.7 (3)
P—C(21)—C(22)	123.6 (2)	P—C(21)—C(26)	117.9 (2)
C(22)—C(21)—C(26)	118.5 (2)	C(21)—C(22)—C(23)	120.4 (2)
C(22)—C(23)—C(24)	120.4 (3)	C(23)—C(24)—C(25)	120.0 (3)
C(24)—C(25)—C(26)	119.9 (3)	C(21)—C(26)—C(25)	120.8 (2)

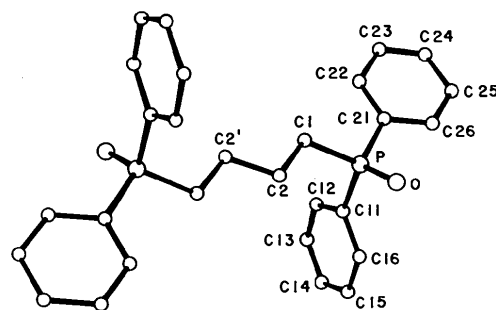


Fig. 1. Perspective view of the molecule showing the atom labeling.

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Related literature. The molecule is sited on a crystallographic center of symmetry which relates one half of the molecule to the other. Related structures have been described by Oliva, Castellano & De Carvalho (1981) and Rivera, Gómez C, Rodulfo de Gil & Suarez (1988).

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Structure of *endo*-(5*R**,6*R**,11*R**,12*S**)-5,6,11,12-Tetrahydro-4,11,12-trimethoxy-9,13,13-trimethyl-5-(triethylsiloxy)-6,10-methano-8(7*H*)-benzocyclodecenone

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Abstract. $C_{27}H_{42}O_5Si$, $M_r = 474.71$, monoclinic, $P2_1/n$, $a = 9.389$ (5), $b = 35.542$ (7), $c = 8.899$ (4) Å, $\beta = 114.25$ (3)°, $V = 2707$ (2) Å³, $Z = 4$, $D_x = 1.164$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.14$ cm⁻¹, $F(000) = 1032$, $T = 298$ K, final $R = 0.049$ for 2926 unique reflections [$I > 3.0\sigma(I)$]. This C-aromatic taxane-like compound contains a C=C double bond [C(9)=C(10)] at the bridgehead site [C(10)] and, consequently, atoms C(8), C(11), C(13) and C(16) bonded to this C=C bond are twisted

from coplanarity. The largest torsion angle, C(8)—C(9)—C(10)—C(11), is -158.3 (3)°.

Experimental. Colorless plate-like crystals grown from hexane. Crystal size $0.40 \times 0.40 \times 0.50$ mm, Rigaku AFC-5R diffractometer, graphite-monochromated Mo $K\alpha$ radiation, ω scan with scan speed $16^\circ \text{ min}^{-1}$ in ω , scan width $(1.34 + 0.35 \tan \theta)^\circ$. Range of indices, $0 < h < 11$, $0 < k < 42$, $-9 < l < 9$ ($2\theta < 50^\circ$). Three standard reflections ($\bar{3}\bar{3}\bar{3}$, $50\bar{5}$, $\bar{4}\bar{1}\bar{6}, 2$) monitored every 100 reflections with random variation of 2.7% over data collection. Lattice-

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parameter determination based on 24 2θ values ($25 < 2\theta < 30^\circ$). 5183 reflections measured, 4877 unique ($R_{\text{int}} = 0.02$); 2926 observed reflections with $I > 3.0\sigma(I)$. No corrections for absorption or extinction. Structure solved by direct methods and refined by full-matrix least squares. The locations of the methyl H atoms of C(16), C(19) and C(20), the ethyl H atoms and the H attached to C(11) were calculated stereochemically and not refined. The other H atoms were obtained from a difference map. Non-H atoms refined with anisotropic thermal parameters, and H atoms with isotropic thermal parameters. $\sum w(|F_o| - |F_c|)^2$ minimized for $w^{-1} = \sigma^2(F_o)$. Final $R = 0.049$,

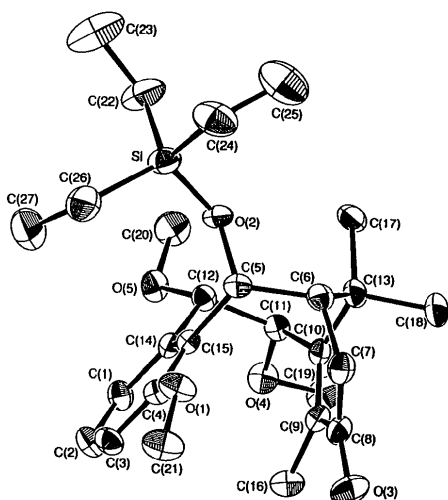


Fig. 1. A perspective view of the molecule (30% probability ellipsoids) with the numbering scheme.

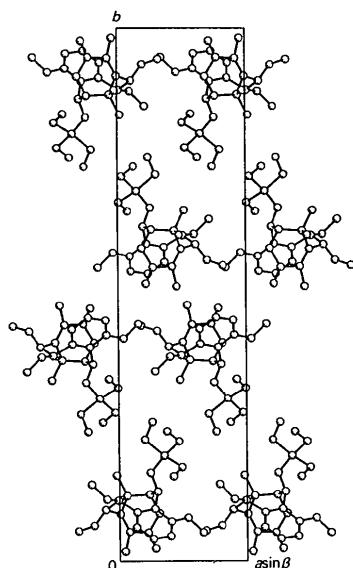


Fig. 2. Crystal structure viewed along the c axis.

Table 1. Final fractional coordinates and equivalent isotropic temperature factors for non-H atoms with *e.s.d.*'s in parentheses

$$B_{\text{eq}} = (\frac{3}{2}\pi^2) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
Si	0.3530 (1)	0.19574 (3)	0.5452 (1)	4.41 (4)
O(1)	0.5393 (2)	0.08087 (6)	0.5377 (3)	4.5 (1)
O(2)	0.2462 (2)	0.16427 (5)	0.4119 (3)	3.6 (1)
O(3)	0.2819 (3)	0.02323 (7)	0.1231 (3)	5.6 (1)
O(4)	-0.1970 (3)	0.06654 (6)	0.2490 (3)	4.2 (1)
O(5)	-0.0971 (2)	0.11552 (6)	0.5019 (3)	4.0 (1)
C(1)	0.1354 (4)	0.0632 (1)	0.6076 (4)	3.6 (1)
C(2)	0.2649 (4)	0.0413 (1)	0.6919 (4)	4.2 (2)
C(3)	0.4002 (4)	0.0462 (1)	0.6684 (4)	3.8 (1)
C(4)	0.4064 (3)	0.07398 (8)	0.5633 (4)	3.2 (1)
C(5)	0.3014 (4)	0.12948 (8)	0.3743 (4)	3.1 (1)
C(6)	0.2389 (4)	0.1240 (1)	0.1843 (4)	3.5 (1)
C(7)	0.3136 (4)	0.0890 (1)	0.1476 (5)	4.1 (2)
C(8)	0.2255 (4)	0.0531 (1)	0.1387 (4)	3.8 (1)
C(9)	0.0780 (4)	0.05461 (9)	0.1660 (4)	3.2 (1)
C(10)	0.0058 (3)	0.08792 (8)	0.1532 (3)	2.9 (1)
C(11)	-0.1071 (4)	0.09642 (9)	0.2314 (4)	3.3 (1)
C(12)	-0.0079 (4)	0.1132 (1)	0.4056 (4)	3.0 (1)
C(13)	0.0602 (4)	0.12178 (9)	0.0836 (4)	3.5 (1)
C(14)	0.1401 (3)	0.09128 (8)	0.5012 (3)	2.8 (1)
C(15)	0.2780 (3)	0.09761 (8)	0.4784 (3)	2.8 (1)
C(16)	0.0422 (4)	0.0179 (1)	0.2272 (5)	4.3 (1)
C(17)	-0.0196 (5)	0.1596 (1)	0.0808 (5)	4.2 (2)
C(18)	0.0217 (6)	0.1140 (1)	-0.1005 (5)	5.0 (2)
C(19)	-0.3207 (4)	0.0565 (1)	0.0952 (5)	5.4 (2)
C(20)	-0.2141 (5)	0.1432 (1)	0.4479 (5)	5.6 (2)
C(21)	0.6577 (5)	0.0531 (1)	0.5860 (8)	5.6 (2)
C(22)	0.2030 (5)	0.2259 (1)	0.5733 (6)	7.4 (2)
C(23)	0.2616 (8)	0.2590 (2)	0.6879 (9)	11.5 (4)
C(24)	0.4710 (5)	0.2227 (1)	0.4512 (6)	7.3 (2)
C(25)	0.3758 (7)	0.2412 (2)	0.2891 (8)	10.3 (3)
C(26)	0.4939 (5)	0.1747 (1)	0.7405 (5)	6.5 (2)
C(27)	0.4253 (7)	0.1578 (1)	0.8516 (6)	9.1 (3)

Table 2. Selected bond lengths (\AA) and angles ($^\circ$) with *e.s.d.*'s in parentheses

Si—O(2)	1.639 (2)	C(2)—C(1)—C(14)	120.5 (3)
O(2)—C(5)	1.432 (4)	C(1)—C(2)—C(3)	120.6 (3)
C(1)—C(2)	1.377 (5)	C(2)—C(3)—C(4)	119.4 (3)
C(1)—C(14)	1.389 (4)	C(3)—C(4)—C(15)	121.7 (3)
C(2)—C(3)	1.380 (5)	C(6)—C(5)—C(15)	118.6 (3)
C(3)—C(4)	1.378 (4)	C(5)—C(6)—C(7)	109.5 (3)
C(4)—C(15)	1.408 (4)	C(5)—C(6)—C(13)	118.1 (3)
C(5)—C(6)	1.558 (4)	C(7)—C(6)—C(13)	108.7 (3)
C(5)—C(15)	1.535 (4)	C(6)—C(7)—C(8)	114.3 (3)
C(6)—C(7)	1.527 (5)	C(7)—C(8)—C(9)	119.0 (3)
C(6)—C(13)	1.546 (5)	C(8)—C(9)—C(10)	119.0 (3)
C(7)—C(8)	1.505 (5)	C(9)—C(10)—C(11)	124.0 (3)
C(8)—C(9)	1.501 (4)	C(9)—C(10)—C(13)	119.0 (3)
C(9)—C(10)	1.346 (4)	C(11)—C(10)—C(13)	116.1 (3)
C(10)—C(11)	1.518 (4)	C(10)—C(11)—C(12)	106.7 (2)
C(10)—C(13)	1.533 (4)	C(11)—C(12)—C(14)	113.4 (2)
C(11)—C(12)	1.561 (4)	C(6)—C(13)—C(10)	105.5 (3)
C(12)—C(14)	1.513 (4)	C(1)—C(14)—C(12)	118.3 (3)
C(14)—C(15)	1.408 (4)	C(1)—C(14)—C(15)	120.1 (3)
		C(12)—C(14)—C(15)	121.5 (3)
		C(4)—C(15)—C(5)	117.4 (3)
		C(4)—C(15)—C(14)	117.6 (3)
		C(5)—C(15)—C(14)	124.9 (3)

$wR = 0.057$, $S = 1.96$ for 366 variables. $(\Delta/\sigma)_{\text{max}} = 0.01$, $\Delta\rho_{\text{max}} = 0.34$, $\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV) and programs used were those from *TEXSAN* (Molecular Structure Corporation, 1985).

The molecular structure and the crystal structure viewed along the c axis are shown in Figs. 1 and 2, respectively. Positional parameters and equivalent

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. C₁₅H₂₀O₃, $M_r = 248.3$, orthorhombic, $C222_1$, $a = 7.5919$ (7), $b = 15.5508$ (7), $c = 22.349$ (3) Å, $V = 2638.5$ (7) Å³, $Z = 8$, $D_x = 1.250$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54184$ 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 BF₃-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 $30 > \theta > 25^\circ$. 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 ($2 < \theta < 75^\circ$) $0 \leq h \leq 9$, $0 \leq k \leq 19$, $0 \leq l \leq 28$ 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_o , using data for which $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o)^2]^{-1}$ 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) × 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