scheme are shown in the *ORTEP*II (Johnson, 1976) drawing in Fig. 1.

Related literature. The isolation of Δ^{8}-1′,2′-dihydro-3,4,3′,4′-bismethylenedioxy-2′-oxo-8.1-neolignan has been reported, along with a partial structure assignment based on NMR data (Green & Wiemer, 1991). For a description of other work on the chemistry of *Piper capense* roots, see Green, Galinis & Wiemer (1991).

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1,1,4-Trimethylsilacyclohexan-4-ol

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Abstract. $C_8H_{18}OSi$, $M_r = 158.32$, monoclinic, $C2/c$, $a = 40.88$ (1), $b = 6.733$ (4), $c = 30.16$ (1) Å, $\beta = 132.08$ (3)°, $V = 6161$ Å^3, $Z = 24$, $D_x = 1.02$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.68$ cm^{-1}, $F(000) = 2112$, $T = 298$ K, $R = 0.059$, $wR = 0.049$ and $S = 1.71$ for 4194 observed reflections. The three independent molecules, two in the more stable conformation with the OH axial and the third with the OH equatorial, form an infinite hydrogen-bonded helix.

Experimental. A colorless prism, 0.20 × 0.25 × 0.30 mm, was obtained by slow evaporation from methanol, and used for data collection on an Enraf–Nonius CAD-4 diffractometer with Mo $K\alpha$ radiation and ω–2θ scans to $2\theta_{\max} = 53°$. 25 reflections with $20 < 2\theta < 26°$ were used for unit-cell determination. Space group was determined from systematic absences (hkl, $h + k$ odd; $h0l$, l odd) and successful refinement. Three standard reflections measured after every 7200 s of X-ray exposure showed no deterioration. For $-49 < h < 38$, $0 < k < 8$, $0 < l < 37$, no redundant data were collected; a total of 6368 unique reflections were measured with 4149 observed [$I > 2\sigma(I)$]. Data were corrected for Lorentz and polarization effects but not for absorption. The structure was solved by direct methods (Sheldrick, 1985). H atoms were located on difference Fourier maps and refined with isotropic thermal parameters; non-H atoms were refined with anisotropic thermal parameters. Refinement was on F; function minimized during refinement was $\sum w(|F_o| - |F_c|)^2$, with $w = 4F_o^2[\sigma^2(I) + (0.04F_o^2)^2]^{-1}$. $R = 0.059$, $wR = 0.049$ and $S = 1.71$ for the observed data and 487 parameters; $(\Delta/\sigma)_{\max}$ in final cycle was less than 2%; the final difference map showed maxima of ± 0.2 e Å^{-3}. Atomic scattering factors and anomalous-dispersion corrections were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations were performed with *SHELX*76 (Sheldrick, 1976).

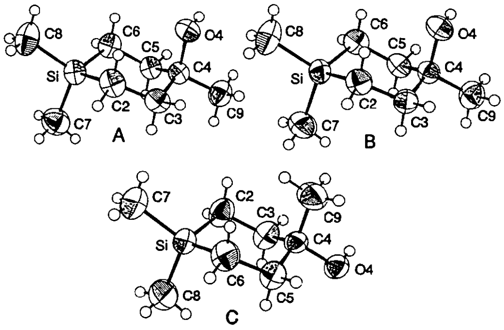

Fig. 1. Perspective view of the three independent molecules with numbering scheme. Thermal ellipsoids are drawn at the 50% probability level.

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Table 1. *Positional and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	U_{eq}
Si(*A*)	0.15883 (03)	0.76408 (11)	0.40996 (3)	0.0466 (4)
O(*A*)	0.1004 (1)	0.9666 (3)	0.2581 (1)	0.050 (1)
C(2*A*)	0.1053 (1)	0.6843 (4)	0.3370 (1)	0.057 (1)
C(3*A*)	0.0728 (1)	0.8564 (4)	0.3024 (1)	0.053 (1)
C(4*A*)	0.0895 (1)	1.0368 (3)	0.2921 (1)	0.043 (1)
C(5*A*)	0.1303 (1)	1.1302 (4)	0.3501 (1)	0.047 (1)
C(6*A*)	0.1709 (1)	0.9958 (4)	0.3896 (1)	0.053 (1)
C(7*A*)	0.1529 (2)	0.8188 (8)	0.4645 (2)	0.079 (2)
C(8*A*)	0.2020 (1)	0.5711 (6)	0.4425 (2)	0.075 (2)
C(9*A*)	0.0529 (1)	1.1910 (5)	0.2555 (2)	0.061 (2)
Si(*B*)	0.04729 (2)	0.49201 (12)	0.43662 (3)	0.0520 (5)
O(*B*)	−0.0709 (1)	0.6501 (3)	0.3211 (1)	0.051 (1)
C(2*B*)	−0.0016 (1)	0.3948 (4)	0.4218 (2)	0.053 (1)
C(3*B*)	−0.0269 (1)	0.5597 (4)	0.4226 (1)	0.049 (1)
C(4*B*)	−0.0415 (1)	0.7334 (3)	0.3804 (1)	0.043 (1)
C(5*B*)	−0.0032 (1)	0.8378 (4)	0.3919 (1)	0.051 (1)
C(6*B*)	0.0241 (1)	0.7085 (5)	0.3854 (2)	0.055 (2)
C(7*B*)	0.0898 (1)	0.5760 (8)	0.5157 (2)	0.080 (2)
C(8*B*)	0.0715 (2)	0.3033 (7)	0.4206 (3)	0.085 (3)
C(9*B*)	−0.0664 (1)	0.8831 (5)	0.3862 (2)	0.065 (2)
Si(*C*)	0.22842 (3)	0.82270 (13)	0.14244 (4)	0.0589 (5)
O(*C*)	0.3771 (1)	0.7993 (3)	0.2758 (1)	0.055 (1)
C(2*C*)	0.2643 (1)	1.0430 (5)	0.1665 (2)	0.072 (2)
C(3*C*)	0.3119 (1)	0.9836 (5)	0.1997 (2)	0.063 (2)
C(4*C*)	0.3337 (1)	0.8459 (4)	0.2528 (1)	0.046 (1)
C(5*C*)	0.3105 (1)	0.6458 (4)	0.2347 (2)	0.057 (2)
C(6*C*)	0.2629 (1)	0.6561 (5)	0.2083 (2)	0.067 (2)
C(7*C*)	0.1760 (2)	0.8932 (10)	0.1223 (3)	0.098 (3)
C(8*C*)	0.2168 (2)	0.7017 (10)	0.0779 (2)	0.107 (3)
C(9*C*)	0.3386 (1)	0.9390 (8)	0.3026 (2)	0.083 (2)

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

Si(*A*)—C(2*A*)	1.861 (4)	C(2*B*)—C(3*B*)	1.527 (5)
Si(*A*)—C(8*A*)	1.856 (4)	C(4*B*)—C(9*B*)	1.524 (6)
Si(*A*)—C(7*A*)	1.854 (8)	C(3*B*)—C(4*B*)	1.524 (5)
Si(*A*)—C(6*A*)	1.860 (4)	C(5*B*)—C(6*B*)	1.528 (6)
O(*A*)—C(4*A*)	1.444 (5)	C(4*B*)—C(5*B*)	1.527 (4)
C(4*A*)—C(5*A*)	1.527 (4)	Si(*C*)—C(2*C*)	1.858 (4)
C(2*A*)—C(3*A*)	1.530 (4)	Si(*C*)—C(8*C*)	1.855 (9)
C(4*A*)—C(9*A*)	1.522 (4)	Si(*C*)—C(7*C*)	1.857 (6)
C(3*A*)—C(4*A*)	1.524 (4)	Si(*C*)—C(6*C*)	1.854 (4)
C(5*A*)—C(6*A*)	1.531 (4)	O(*C*)—C(4*C*)	1.442 (3)
Si(*B*)—C(2*B*)	1.850 (4)	C(2*C*)—C(3*C*)	1.529 (5)
Si(*B*)—C(8*B*)	1.861 (7)	C(4*C*)—C(9*C*)	1.511 (8)
Si(*B*)—C(7*B*)	1.863 (5)	C(4*C*)—C(5*C*)	1.522 (4)
Si(*B*)—C(6*B*)	1.856 (4)	C(3*C*)—C(4*C*)	1.519 (5)
O(*B*)—C(4*B*)	1.442 (4)	C(5*C*)—C(6*C*)	1.533 (5)

C(2*A*)—Si(*A*)—C(6*A*)	102.4 (2)	C(2*A*)—Si(*A*)—C(7*A*)	110.9 (2)
C(2*A*)—Si(*A*)—C(8*A*)	111.8 (2)	C(6*A*)—Si(*A*)—C(7*A*)	110.3 (2)
C(6*A*)—Si(*A*)—C(8*A*)	113.1 (2)	C(7*A*)—Si(*A*)—C(8*A*)	108.2 (3)
Si(*A*)—C(2*A*)—C(3*A*)	112.6 (2)	C(2*A*)—C(3*A*)—C(4*A*)	116.1 (3)
O(*A*)—C(4*A*)—C(3*A*)	106.1 (2)	O(*A*)—C(4*A*)—C(5*A*)	109.0 (2)
O(*A*)—C(4*A*)—C(9*A*)	109.2 (3)	C(3*A*)—C(4*A*)—C(5*A*)	113.0 (3)
C(3*A*)—C(4*A*)—C(9*A*)	109.6 (3)	C(5*A*)—C(4*A*)—C(9*A*)	109.8 (2)
C(4*A*)—C(5*A*)—C(6*A*)	115.6 (2)	Si(*A*)—C(6*A*)—C(5*A*)	111.4 (2)
C(2*B*)—Si(*B*)—C(6*B*)	102.1 (2)	C(2*B*)—Si(*B*)—C(7*B*)	110.3 (2)
C(2*B*)—Si(*B*)—C(8*B*)	111.8 (2)	C(6*B*)—Si(*B*)—C(7*B*)	110.2 (2)
C(6*B*)—Si(*B*)—C(8*B*)	112.6 (3)	C(7*B*)—Si(*B*)—C(8*B*)	109.7 (3)
Si(*B*)—C(2*B*)—C(3*B*)	111.8 (2)	C(2*B*)—C(3*B*)—C(4*B*)	115.9 (4)
O(*B*)—C(4*B*)—C(3*B*)	105.8 (2)	O(*B*)—C(4*B*)—C(5*B*)	109.6 (3)
O(*B*)—C(4*B*)—C(9*B*)	109.5 (3)	C(3*B*)—C(4*B*)—C(5*B*)	113.3 (2)
C(3*B*)—C(4*B*)—C(9*B*)	109.3 (4)	C(5*B*)—C(4*B*)—C(9*B*)	109.3 (2)
C(4*B*)—C(5*B*)—C(6*B*)	115.7 (2)	Si(*B*)—C(6*B*)—C(5*B*)	110.5 (3)
C(2*C*)—Si(*C*)—C(6*C*)	103.3 (2)	C(2*C*)—Si(*C*)—C(8*C*)	109.9 (3)
C(2*C*)—Si(*C*)—C(7*C*)	111.1 (2)	C(6*C*)—Si(*C*)—C(8*C*)	109.6 (3)
C(6*C*)—Si(*C*)—C(7*C*)	112.8 (3)	C(8*C*)—Si(*C*)—C(7*C*)	110.0 (3)
Si(*C*)—C(2*C*)—C(3*C*)	111.9 (2)	C(2*C*)—C(3*C*)—C(4*C*)	116.0 (4)
O(*C*)—C(4*C*)—C(3*C*)	107.6 (3)	O(*C*)—C(4*C*)—C(5*C*)	104.5 (2)
O(*C*)—C(4*C*)—C(9*C*)	108.2 (3)	C(3*C*)—C(4*C*)—C(5*C*)	112.2 (3)
C(3*C*)—C(4*C*)—C(9*C*)	112.8 (3)	C(5*C*)—C(4*C*)—C(9*C*)	111.1 (4)
C(4*C*)—C(5*C*)—C(6*C*)	114.5 (3)	Si(*C*)—C(6*C*)—C(5*C*)	111.9 (3)

Si(*A*)—C(2*A*)—C(3*A*)—C(4*A*)	53.6 (5)	Si(*A*)—C(6*A*)—C(5*A*)—C(4*A*)	−57.2 (4)
O(*A*)—C(4*A*)—C(3*A*)—C(2*A*)	60.5 (4)	O(*A*)—C(4*A*)—C(5*A*)—C(6*A*)	−56.5 (4)
C(2*A*)—Si(*A*)—C(6*A*)—C(5*A*)	46.1 (3)	C(2*A*)—C(3*A*)—C(4*A*)—C(5*A*)	−59.0 (4)
C(2*A*)—C(3*A*)—C(4*A*)—C(9*A*)	178.2 (4)	C(3*A*)—C(2*A*)—Si(*A*)—C(6*A*)	−44.6 (4)
C(3*A*)—C(2*A*)—Si(*A*)—C(7*A*)	73.1 (4)	C(3*A*)—C(2*A*)—Si(*A*)—C(8*A*)	−166.0 (3)
C(3*A*)—C(4*A*)—C(5*A*)—C(6*A*)	61.2 (4)	C(5*A*)—C(6*A*)—Si(*A*)—C(7*A*)	−72.0 (3)
C(5*A*)—C(6*A*)—Si(*A*)—C(8*A*)	166.6 (4)	C(6*A*)—C(5*A*)—C(4*A*)—C(9*A*)	−176.1 (4)
Si(*B*)—C(2*B*)—C(3*B*)—C(4*B*)	55.3 (4)	Si(*B*)—C(6*B*)—C(5*B*)—C(4*B*)	−57.7 (4)
O(*B*)—C(4*B*)—C(3*B*)—C(2*B*)	61.7 (3)	O(*B*)—C(4*B*)—C(5*B*)—C(6*B*)	−57.8 (4)
C(2*B*)—Si(*B*)—C(6*B*)—C(5*B*)	48.7 (3)	C(2*B*)—C(3*B*)—C(4*B*)—C(5*B*)	−58.3 (4)
C(2*B*)—C(3*B*)—C(4*B*)—C(9*B*)	179.6 (3)	C(3*B*)—C(2*B*)—Si(*B*)—C(6*B*)	−47.8 (4)
C(3*B*)—C(2*B*)—Si(*B*)—C(7*B*)	69.2 (4)	C(3*B*)—C(2*B*)—Si(*B*)—C(8*B*)	−168.4 (4)
C(3*B*)—C(4*B*)—C(5*B*)—C(6*B*)	60.0 (5)	C(5*B*)—C(6*B*)—Si(*B*)—C(7*B*)	−68.5 (3)
C(5*B*)—C(6*B*)—Si(*B*)—C(8*B*)	168.7 (3)	C(6*B*)—C(5*B*)—C(4*B*)—C(9*B*)	−177.8 (4)
Si(*C*)—C(2*C*)—C(3*C*)—C(4*C*)	−54.2 (5)	Si(*C*)—C(6*C*)—C(5*C*)—C(4*C*)	56.8 (5)
O(*C*)—C(4*C*)—C(3*C*)—C(2*C*)	177.0 (3)	O(*C*)—C(4*C*)—C(5*C*)—C(6*C*)	−179.9 (4)
C(2*C*)—Si(*C*)—C(6*C*)—C(5*C*)	−44.2 (4)	C(2*C*)—C(3*C*)—C(4*C*)—C(5*C*)	62.6 (5)
C(2*C*)—C(3*C*)—C(4*C*)—C(9*C*)	−63.8 (4)	C(3*C*)—C(2*C*)—Si(*C*)—C(6*C*)	42.6 (5)
C(3*C*)—C(2*C*)—Si(*C*)—C(8*C*)	−74.3 (5)	C(3*C*)—C(2*C*)—Si(*C*)—C(7*C*)	163.7 (5)
C(3*C*)—C(4*C*)—C(5*C*)—C(6*C*)	−63.6 (5)	C(5*C*)—C(6*C*)—Si(*C*)—C(8*C*)	72.9 (4)
C(5*C*)—C(6*C*)—Si(*C*)—C(7*C*)	−164.2 (3)	C(6*C*)—C(5*C*)—C(4*C*)—C(9*C*)	63.7 (5)

Perspective drawings of the three independent molecules with the numbering scheme are given in Fig. 1 (Johnson, 1976). Final positional parameters for the non-H atoms are given in Table 1.* Bond lengths and angles and conformational angles are given in Table 2. Fig. 2 (Johnson, 1976) is a stereoview of the hydrogen-bonded helical structure generated by translation of the three crystallographically independent molecules along the short *b* axis. The O···O distances in the helix are $A \to B$ 2.799 (5), $B \to C$ 2.841 (5) and $A \to C$ 2.858 (5) Å.

Calculations of conformational energy were performed on the carbinol with the program *MMX* (Serena Software, 1989) and are in excellent agreement with the 2:1 preference for the Me_{eq}, OH_{ax} (molecules *A* and *B*) over the less stable Me_{ax}, OH_{eq} (molecule *C*) conformation ($\Delta MMXE = 4$ kJ mol^{-1}). The average vicinal proton coupling constants, $^3J_{trans} = 9$ and $^3J_{cis} = 4$ Hz, which were determined from the 300 MHz NMR data on the title compound, were virtually identical to those calculated values from the *MMX*-minimized structures for both conformations. The use of Lambert's relationship (Lambert, 1971) from the measured NMR coupling constants results in a calculated torsional dihedral angle of 58.5°; the overall average crystallographic value is 55.8° and the overall *MMX*-calculated value is 56.4°. The good agreement between these calculated and experimental values for solid-state and solution environments for the silacyclohexyl system provides a firmer basis for understanding the significant conformation features of this interesting heterocyclic system.

Related literature. The title compound (m.p. 316–318 K) was prepared in 84% yield from the addition of 1,1-dimethyl-1-silacyclohexan-4-one to methyllithium in diethyl ether at 351 K followed by an aqueous NH_4Cl quench at 298 K (Soderquist & Negron, 1989). Analytical and spectroscopic data

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55679 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GR0205]

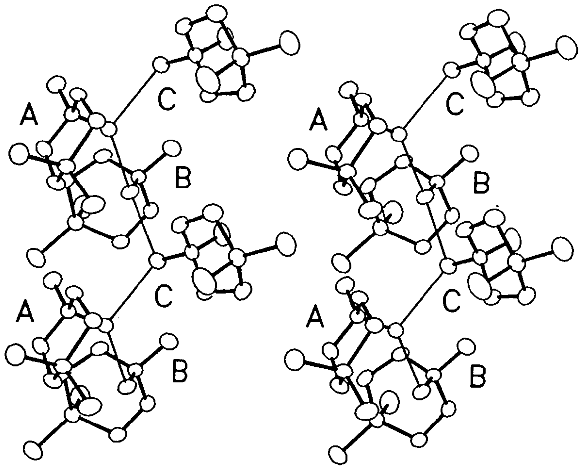

Fig. 2. Stereoview of the hydrogen-bonded structure viewed normal to the *b* axis.

were obtained on the product which were wholly consistent with the assigned structure.

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Structure of Galphimine B

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Abstract. (4*R*)-Trihydroxy-13α-methoxycarbonyl-30-nor-3,4-seco-7α,18β-fridela-1,20-dien-3,24-olide methylene chloride solvate, $C_{30}H_{44}O_7.0.898CH_2Cl_2$, $M_r = 601.6$, orthorhombic, $P2_12_12_1$, $a = 9.797\,(4)$, $b = 15.039\,(7)$, $c = 20.135\,(8)$ Å, $V = 2966.5\,(8)$ Å^3, $Z = 4$, $D_x = 1.347$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 23.66$ cm^{-1}, $F(000) = 1288$, $T = 298$ K, $R = 0.062$ for 2004 reflections with $F > 3\sigma(F)$. In the pentacyclic molecule, rings *C* and *D* adopt a chair conformation while ring *E* shows a half-chair conformation (ring junctions: *C*/*D*, *trans*; *D*/*E*, *cis*). The seven-membered ring *A* and six-membered ring *B* are considerably distorted (twist and monoplanar conformations, respectively) which is reflected in the pseudo-*trans* *A*/*B* ring junction [torsion angles $-11.3\,(3)$ and $1.3\,(3)°$] Intramolecular O(3)—H(3)···O(4) [*D*—H 1.04 (6), H···*A* 1.72 (6), *D*···*A* 2.720 (8) Å, *D*—H···*A* 160 (1)°] and intermolecular O(4)—H(4*a*)···O(3) ($x - 0.5$, $1.5 - y$, $2 - z$) [*D*—H 0.77 (7), H···*A* 2.26 (7), *D*···*A* 3.005 (8) Å, *D*—H···*A* 165 (1)°] hydrogen bonds stabilize the molecules in the crystal.

Experimental. The title compound was isolated from aerial parts of *Galphymia glauca* (*Cav.*) Kuntze. Crystallization from methanol–methylene chloride yielded a colorless single crystal of dimensions 0.26 × 0.30 × 0.38 mm. The unit-cell parameters were refined from least-squares analysis of 2θ values of 25 reflections from $7.35 < 2\theta < 23.93°$. Intensities for 2183 reflections (2152 unique, $R_{\text{int}} = 0.029$) having $3 < 2\theta < 110°$ and $0 < h < 10$, $0 < k < 15$, $0 < l < 21$, were measured on a Nicolet *P*3*F* diffractometer

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