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***rac*-1,1,4-Trimethyl-5-trimethylsilyl-1,3,3a,4,6,7,8,9-octahydro-1-silaphtho[1,2-*c*]furan at 153 K**

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Abstract

The crystal structure of *rac*-1,1,4-trimethyl-5-trimethylsilyl-1,3,3a,4,6,7,8,9-octahydro-1-silaphtho[1,2-*c*]furan, C₁₇H₃₀OSi₂, has been determined at low temperature.

Comment

The title compound, (2), was obtained in 53% yield via an intramolecular cobalt-mediated [2 + 2 + 2] cycloaddition of the alkenediyne (1) and subsequent oxidative demetallation of the resulting cobalt–cyclohexadiene complex. Further details will be published elsewhere (Eckenberg & Groth, 1995). The reaction proceeded in a stereospecific manner with regard to the geometry of the double bond in compound (1). The X-ray structure analysis was performed in order to establish the relative stereochemistry at atoms C3a and C4, and it confirmed our previous assignments based on NMR techniques (Eckenberg, 1992). The five-membered ring (Si1, O2, C3, C3a, C3b) adopts an envelope conformation. The first six-membered ring (C3b, C3a, C4, C5, C9b, C9a) adopts a twist conformation, whereas the second six-membered ring (C9a, C9b, C6, C7, C8, C9) is in a chair conformation. All bond lengths and angles in compound (2) are in the expected ranges.

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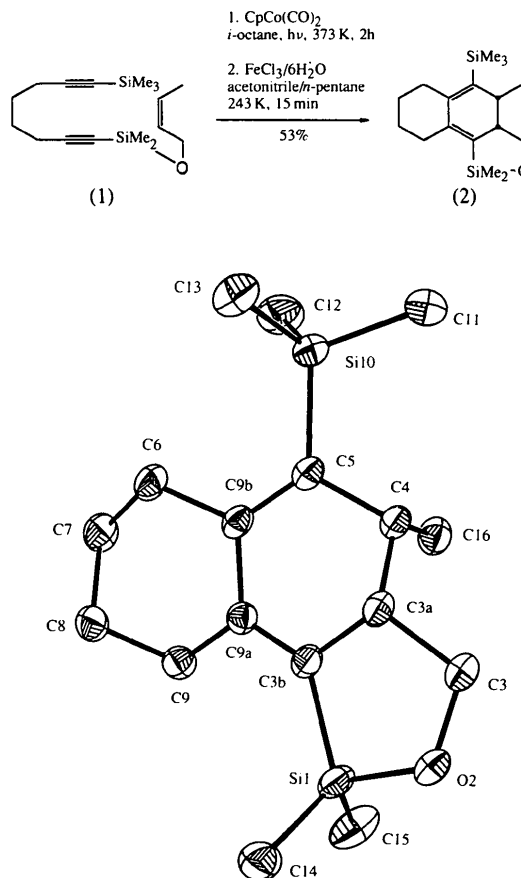


Fig. 1. Crystal structure of compound (2) showing 50% probability displacement ellipsoids. The H atoms are omitted for clarity.

Experimental

The compound was crystallized from a mixture of diethyl ether and pentane at room temperature.

Crystal data

C₁₇H₃₀OSi₂
M_r = 306.59

Mo *K*α radiation
 λ = 0.71073 Å

Orthorhombic	Cell parameters from 52 reflections	C3b—C9a	1.346 (3)	Si10—C13	1.867 (3)
<i>Pccn</i>	$\theta = 10\text{--}12.5^\circ$	C4—C16	1.532 (3)	Si10—C11	1.869 (3)
$a = 17.721 (2) \text{ \AA}$	$\mu = 0.189 \text{ mm}^{-1}$	C4—C5	1.549 (3)	Si10—C12	1.869 (3)
$b = 19.714 (2) \text{ \AA}$	$T = 153 (2) \text{ K}$	O2—Si1—C15	112.72 (12)	C9b—C5—Si10	124.5 (2)
$c = 10.4900 (10) \text{ \AA}$	Block	O2—Si1—C14	107.85 (10)	C4—C5—Si10	117.7 (2)
$V = 3664.7 (7) \text{ \AA}^3$	0.7 × 0.7 × 0.3 mm	C15—Si1—C14	109.92 (14)	C7—C6—C9b	114.2 (2)
$Z = 8$	Colourless	O2—Si1—C3b	94.65 (9)	C8—C7—C6	110.6 (2)
$D_x = 1.111 \text{ Mg m}^{-3}$		C15—Si1—C3b	114.06 (12)	C7—C8—C9	109.3 (2)
		C14—Si1—C3b	116.67 (11)	C9a—C9—C8	113.8 (2)
		C3—O2—Si1	113.54 (13)	C3b—C9a—C9b	119.5 (2)
		O2—C3—C3a	109.8 (2)	C3b—C9a—C9	121.8 (2)
		C3b—C3a—C4	110.9 (2)	C9b—C9a—C9	118.7 (2)
		C3b—C3a—C3	107.9 (2)	C5—C9b—C9a	120.9 (2)
		C4—C3a—C3	116.1 (2)	C5—C9b—C6	122.7 (2)
		C9a—C3b—C3a	118.8 (2)	C9a—C9b—C6	116.3 (2)
		C9a—C3b—Si1	134.2 (2)	C13—Si10—C11	107.56 (13)
		C3a—C3b—Si1	106.98 (15)	C13—Si10—C12	110.60 (13)
		C3a—C4—C16	114.8 (2)	C11—Si10—C12	106.45 (13)
		C3a—C4—C5	108.9 (2)	C13—Si10—C5	110.17 (11)
		C16—C4—C5	108.8 (2)	C11—Si10—C5	109.62 (11)
		C9b—C5—C4	117.7 (2)	C12—Si10—C5	112.27 (12)

Data collection

Stoe Siemens four-circle diffractometer	$R_{\text{int}} = 0.0118$
Profile data from $2\theta/\omega$ scans	$\theta_{\text{max}} = 22.49^\circ$
Absorption correction: none	$h = -3 \rightarrow 19$
2412 measured reflections	$k = -21 \rightarrow 21$
2380 independent reflections	$l = 0 \rightarrow 11$
2013 observed reflections	3 standard reflections
$[I > 2\sigma(I)]$	frequency: 90 min
	intensity decay: none

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\text{max}} = 0.001$
$R(F) = 0.0361$	$\Delta\rho_{\text{max}} = 0.226 \text{ e \AA}^{-3}$
$wR(F^2) = 0.0927$	$\Delta\rho_{\text{min}} = -0.234 \text{ e \AA}^{-3}$
$S = 1.068$	Atomic scattering factors
2380 reflections	from <i>International Tables for Crystallography</i> (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
200 parameters	
$w = 1/[\sigma^2(F_o^2) + (0.0399P)^2 + 2.6065P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	U_{eq}
Si1	0.40329 (4)	0.17510 (3)	0.19290 (6)	0.0316 (2)
O2	0.46096 (9)	0.12503 (7)	0.2753 (2)	0.0369 (4)
C3	0.53341 (14)	0.15477 (11)	0.2994 (3)	0.0355 (6)
C3a	0.52631 (12)	0.23318 (10)	0.3027 (2)	0.0278 (5)
C3b	0.46007 (12)	0.25300 (11)	0.2191 (2)	0.0264 (5)
C4	0.59670 (13)	0.27288 (10)	0.2664 (2)	0.0273 (5)
C5	0.57796 (13)	0.34962 (11)	0.2684 (2)	0.0262 (5)
C6	0.48281 (14)	0.44264 (11)	0.2292 (2)	0.0344 (6)
C7	0.42793 (14)	0.46084 (12)	0.1237 (3)	0.0404 (7)
C8	0.36215 (13)	0.41213 (11)	0.1224 (3)	0.0369 (6)
C9	0.39092 (13)	0.34090 (11)	0.0946 (2)	0.0301 (6)
C9a	0.45307 (12)	0.31816 (11)	0.1825 (2)	0.0250 (5)
C9b	0.50901 (13)	0.36899 (11)	0.2272 (2)	0.0258 (5)
Si10	0.65387 (4)	0.40959 (3)	0.32329 (6)	0.0319 (2)
C11	0.74046 (14)	0.36055 (13)	0.3663 (3)	0.0452 (7)
C12	0.6825 (2)	0.47055 (14)	0.1957 (3)	0.0525 (8)
C13	0.6224 (2)	0.45607 (13)	0.4690 (3)	0.0506 (8)
C14	0.30940 (14)	0.17212 (13)	0.2700 (3)	0.0444 (7)
C15	0.3952 (2)	0.14975 (13)	0.0237 (3)	0.0555 (8)
C16	0.62998 (13)	0.25485 (13)	0.1360 (2)	0.0349 (6)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Si1—O2	1.663 (2)	C5—C9b	1.351 (3)
Si1—C15	1.850 (3)	C5—Si10	1.881 (2)
Si1—C14	1.851 (3)	C6—C7	1.516 (3)
Si1—C3b	1.856 (2)	C6—C9b	1.524 (3)
O2—C3	1.434 (3)	C7—C8	1.510 (3)
C3—C3a	1.551 (3)	C8—C9	1.522 (3)
C3a—C3b	1.517 (3)	C9—C9a	1.504 (3)
C3a—C4	1.521 (3)	C9a—C9b	1.485 (3)

Intensities were measured with a variable scan speed using a learnt-profile method (Clegg, 1981). All non-H atoms were refined anisotropically. H atoms were refined using a riding model. The C—H bond distances were refined with distance restraints for chemically equivalent H atoms. The isotropic displacement parameters were set to 1.2 times (1.5 times for CH₃ groups) the equivalent displacement parameter of the atom to which they were attached.

Data collection: *DIF4* (Stoe & Cie 1988a). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie 1988b). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1100). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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