References

Bunning, T. J. (1992). PhD dissertation, Univ. of Connecticut, USA.

Bunning, T. J., Klei, H. E., Samulski, E. T., Adams, W. W. & Crane, R. L. (1993). *Mol. Cryst. Liq. Cryst.* **231**, 163–174.

Bunning, T. J., Klei, H. E., Samulski, E. T., Crane, R. L. & Linville, R. J. (1991). *Liq. Cryst.* **10**, 445–456.

Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Polidori, G., Spagna, R. & Viterbo, D. (1989). *J. Appl. Cryst.* **22**, 389–393.

Enraf–Nonius (1989). *CAD-4 Software.* Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis.* Enraf–Nonius, Delft, The Netherlands.

Gresham, K. D., McHugh, C. M., Bunning, T. J., Klei, H. E., Samulski, E. T. & Crane, R. L. (1993). *Am. Chem. Soc. Polym. Prep.* **34**, 594–595.

Guerina, N. G. & Craven, B. M. (1979). *J. Chem. Soc. Perkin Trans.* 2, pp. 1414–1419.

Johnson, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Pattabhi, V. & Craven, B. M. (1979). *J. Lipid Res.* **20**, 753–759.

Reinitzer, F. (1888). *Monatsh. Chem.* **9**, 421–441.

Sato, T. & Yano, S. (1987). *Mol. Cryst. Liq. Cryst.* **144**, 179–184.

Sawzik, P. & Craven, B. M. (1979a). *Acta Cryst.* B35, 895–901.

Sawzik, P. & Craven, B. M. (1979b) *Liquid Crystals*, edited by S. Chandresekhar, pp. 171–178. Bangalore: Heyden.

Sawzik, P. & Craven, B. M. (1980a). *Acta Cryst.* B36, 3027–3033.

Sawzik, P. & Craven, B. M. (1980b). *Acta Cryst.* B36, 215–218.

Shibaev, V. P. & Freidzon, Ya. S. (1989). *Side Chain Liquid Crystal Polymers*, edited by C. B. McArdle, pp. 260–286. Glasgow: Blackie.

dition 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.
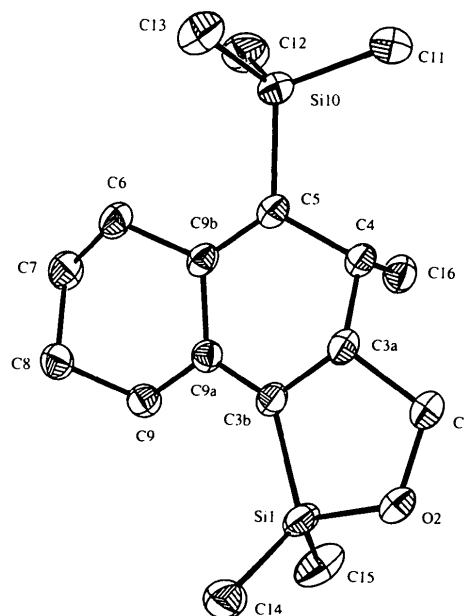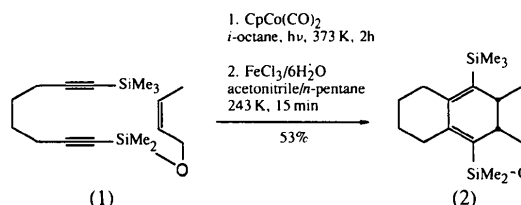

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rac-1,1,4-Trimethyl-5-trimethylsilyl-1,3,3a,4,6,7,8,9-octahydro-1-sila-naphtho[1,2-c]furan at 153 K

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Abstract

The crystal structure of rac-1,1,4-trimethyl-5-trimethyl-silyl-1,3,3a,4,6,7,8,9-octahydro-1-silanaphtho[1,2-c]-furan, $C_{17}H_{30}OSi_2$, has been determined at low temperature.

Comment

The title compound, (2), was obtained in 53% yield *via* an intramolecular cobalt-mediated [2 + 2 + 2] cycload-

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

$C_{17}H_{30}OSi_2$	Mo $K\alpha$ radiation
$M_r = 306.59$	$\lambda = 0.71073$ Å

Orthorhombic
Pccn
$a = 17.721\,(2)$ Å
$b = 19.714\,(2)$ Å
$c = 10.4900\,(10)$ Å
$V = 3664.7\,(7)$ Å^3
$Z = 8$
$D_x = 1.111$ Mg m^{-3}

Cell parameters from 52
reflections
$\theta = 10–12.5°$
$\mu = 0.189$ mm^{-1}
$T = 153\,(2)$ K
Block
$0.7 \times 0.7 \times 0.3$ mm
Colourless

Data collection

Stoe Siemens four-circle
diffractometer
Profile data from $2\theta/\omega$ scans
Absorption correction:
none
2412 measured reflections
2380 independent reflections
2013 observed reflections
$[I > 2\sigma(I)]$

$R_{int} = 0.0118$
$\theta_{max} = 22.49°$
$h = -3 \rightarrow 19$
$k = -21 \rightarrow 21$
$l = 0 \rightarrow 11$
3 standard reflections
frequency: 90 min
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.0361$
$wR(F^2) = 0.0927$
$S = 1.068$
2380 reflections
200 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0399P)^2 + 2.6065P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.226$ e Å^{-3}
$\Delta\rho_{min} = -0.234$ e Å^{-3}
Atomic scattering factors
from *International Tables
for Crystallography* (1992,
Vol. C, Tables 4.2.6.8 and
6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent
isotropic displacement 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}
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* (Å, °)

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)

C3b—C9a	1.346 (3)	Si10—C13	1.867 (3)
C4—C16	1.532 (3)	Si10—C11	1.869 (3)
C4—C5	1.549 (3)	Si10—C12	1.869 (3)
O2—Si1—C15	112.72 (12)	C9b—C5—Si10	124.5 (2)
O2—Si1—C14	107.85 (10)	C4—C5—Si10	117.7 (2)
C15—Si1—C14	109.92 (14)	C7—C6—C9b	114.2 (2)
O2—Si1—C3b	94.65 (9)	C8—C7—C6	110.6 (2)
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)

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 CH$_3$ groups) the equivalent displacement parameter of the atom to which they were attached.

Data collection: *DIF*4 (Stoe & Cie 1988a). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie 1988b). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *SHELXL*93.

References

Clegg, W. (1981). *Acta Cryst.* A**37**, 22–28.
Eckenberg, P. (1992). PhD thesis, Univ. of Göttingen, Germany.
Eckenberg, P. & Groth, U. (1995). In preparation.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.3 for Siemens Crystallographic Research Systems. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for Crystal Structure Refinement*. Univ. of Göttingen, Germany.
Stoe & Cie (1988a). *DIF*4. *Diffractometer Control Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1988b). *REDU*4. *Data Reduction Program*. Version 6.2. Stoe & Cie, Darmstadt, Germany.