Oxidative Desilylation of a 5,6:7,8-Bis(dimethylsilyldioxy)[4]radialene

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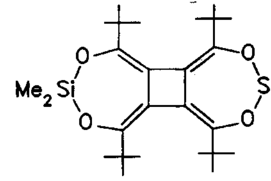
The title [4]radialene **1** reacts with *N*-methyltriazolinedione or with the iodine(III) salt $PhI^+O^+IPh \cdot 2\ BF_4^-$ in CH_2Cl_2 by oxidative elimination of only one dimethylsilyl unit to form the functionalized 3,4-bis(alkylidene)-1-cyclobutene **2** or its hydrolysis products, (*Z*)-**4** and (*E*)-**4**. When the iodonium salt is used in CCl_4, rearrangement of the radialene framework occurs, and the bicyclic fluorodimethylsilyl enol ether **9** is obtained. An X-ray crystal structure analysis of **9** indicates a weak coordination between a carbonyl oxygen atom and a silyl group.

Due to their unusual topology, radialenes have attracted the attention of chemists for some time. Recent years have seen a renewed interest in these molecules, both because of new synthetic methodology and in respect of their potential use for novel organic materials[1].

The chemistry of the radialenes on the whole has certainly not yet been studied in depth. This is especially true for functionalized radialenes. We have recently described the synthesis as well as the acid- and base-catalyzed hydrolysis of the 5,6:7,8-bis(dimethylsilyldioxy)[4]radialene **1**[2,3], which represents one of the few known electron-rich radialenes.



1

In the present paper, we describe additional chemistry of **1** that is based on the presence of two electron-rich 1,3-diene subunits in **1**. One of the appealing aspects of a [4]radialene is the transformation into a cyclobutadiene by simultaneous or subsequent 1,4-addition (including [4 + 2] cycloaddition) to both of its semicyclic 1,3-diene systems. However, all previous attempts have been unsuccessful; for example, the parent [4]radialene[4] and octamethyl[4]radialene[5] can be converted by one such transformation into a 3,4-dimethylene-1-cyclobutene, but the introduction of the second double bond into the four-membered ring cannot be effected. The conjugated silyl enol ether functions in **1** have now offered the opportunity to tackle this problem again.

Results

The reaction of radialene **1** with two equivalents of 4-methyl-1,2,4-triazoline-3,5-dione (MTAD) in boiling toluene produced the cyclobuta[*e*][1,3,2]dioxasilepine **2** in 60% yield as a result of oxidative desilylation. The constitution of **2** was readily derived from its mass spectrum in combination with the ^1H- and ^{13}C-NMR spectra. The fate of MTAD in this reaction was not fully established; however, spectroscopic and analytical data suggest that the barely soluble reaction residue mainly consisted of 4-methylurazole[7,8] and the thermal decomposition product of MTAD, 3,7-dimethyl-1,3,5-triazabicyclo[3.3.0]octane-2,4,6,8-tetrone[8]. Although MTAD is one of the most powerful electron-poor dienophiles for the Diels-Alder reaction, it is not surprising that a [2 + 4] cycloadditon with **1** does not occur: First, the diene subunits of **1** are far from being planar, and their termini are sterically shielded by the *tert*-butyl groups[2]; second, the triazolinediones have a tendency to behave as electrophiles towards electron-rich double bonds[6]. We consider their electrophilic nature to be also responsible for the transformation **1** ⟶ **2** (see below).

Repetition of the oxidative desilylation step that transforms **1** into **2** would give rise to tetrapivaloylcyclobutadiene. Obviously, MTAD is not able to perform this step; even with four equivalents of MTAD, only **2** could be isolated. We therefore turned our attention to other reagents which have been reported to effect the *inter*molecular C–C coupling of two siloxyalkene molecules combined with desilylation, thus providing 1,4-dicarbonyl compounds. The twofold *intra*molecular version of this process would transform radialene **1** into tetrapivaloylcyclobutadiene. Unfortunately, this goal was not achieved, since only inseparable product mixtures resulted from the reaction of **1** with Ag_2O in DMSO[9] or with cerium(IV) ammonium nitrate[10], and the novel hypervalent iodine reagent $PhI^+O^+IPh \cdot 2\ BF_4^-$ (**3**)[11] effected only one intramolecular silyl enol ether coupling in **1**.

From the reaction of **1** with one equivalent of **3** in dichloromethane, cyclobutene (*Z*)-**4** was the only product isolated after aqueous workup. With two equivalents of **3**, only (*E*)-**4** was obtained. Obviously, the cyclobutenes constitute the products of hydrolysis of **2**. In fact, experiments conducted in an NMR tube showed that acidic hydrolysis of **2**

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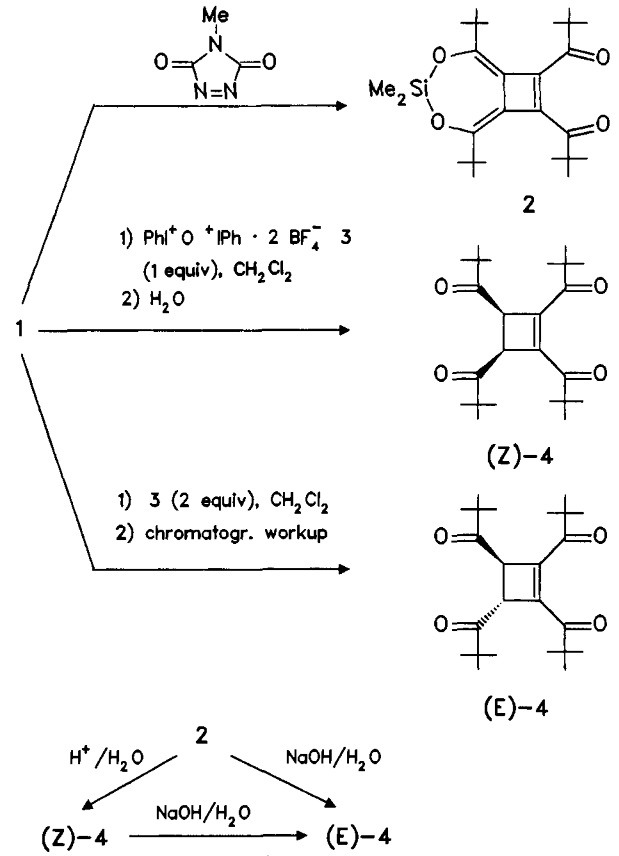
[in $CDCl_3$, addition of 10% (v/v) conc. HCl] leads to (*Z*)-**4**, whereas base-catalyzed hydrolysis [treatment with 10% (v/v) of 2.5 M NaOH] gives rise to (*E*)-**4**.

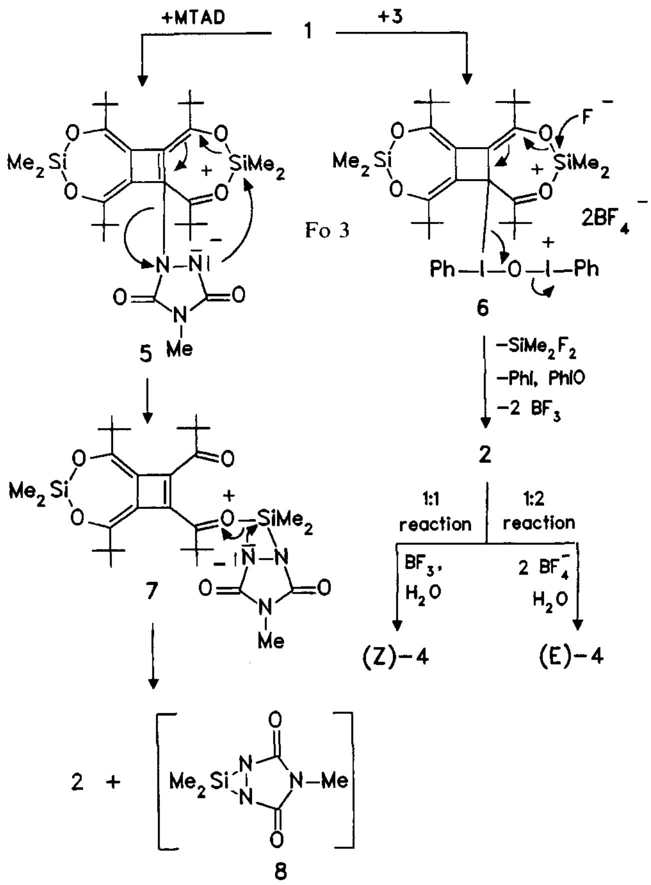
The stereochemical assignment of (*Z*)- and (*E*)-**4** could not be deduced from their very similar ^1H- and ^{13}C-NMR spectra. It became evident, however, by treating both cyclobutenes with dilute aqueous NaOH. This manipulation epimerizes (*Z*)-**4** to the thermodynamically more stable *E*-isomer, whereas it does not alter (*E*)-**4**.



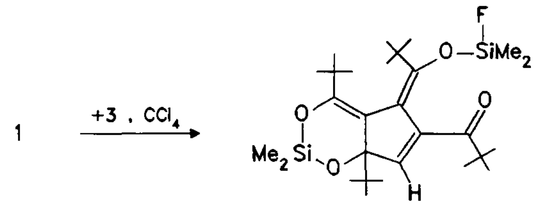
Obviously, the hypervalent iodine salt **3** as well as MTAD are both unable to perform more than one oxidative desilylation step on radialene **1**. These results again demonstrate the high reaction barrier which prevents the transformation of a 3,4-dimethylenecyclobutene into a cyclobutadiene.

In mechanistic terms, the transformations of **1** by MTAD and **3** could proceed as proposed in Scheme 1, with electrophilic attack of the reagents as the initial step leading to **5** and **6**, resp. In the MTAD reaction, the rearrangement **5** → **7** and elimination of **8** from **7** could account for the formation of **2**. 4-Methylurazole, found in the reaction residue, may be the hydrolysis product of **8** which was not detected directly. The transformation of **6** into **2** occurs in fluoride-induced desilylation. With equimolar amounts of **1** and **3**, the 1,3,2-dioxasilepine **2** formed as an intermediate is subsequently hydrolyzed with BF_3 assistance that generates (*Z*)-**4**. The 1:2 reaction, on the other hand, provides an excess of BF_4^- ions, and (*E*)-**4** is formed by F^--mediated desilylation via an enolate.

Scheme 1

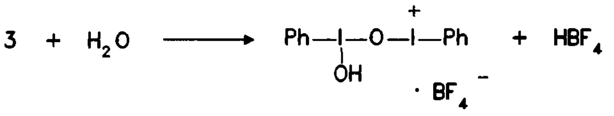


When the reaction of **1** with the bis(iodonium) salt **3** was carried out in CCl_4 rather than CH_2Cl_2, the bicyclic compound **9** bearing a semicyclic fluorodimethylsilyl enol ether function was unexpectedly isolated as the only product, regardless of using one or two equivalents of **3** (39 and 75% yield, resp.). The identity of **9** was established by X-ray diffraction (see below).



9

Evidently, the formation of **9** from **1** merely represents the formal addition of HF to the radialene, rather than an oxidative desilylation as described above. Since the hydrogen atom incorporated into **9** cannot be provided by the solvent, the reagent must be its source. In fact, the preparation of **3** according to the published procedure does not furnish a pure product, and its decomposition point can vary over an unusually broad range[11]. Depending on the contact of **3** with water added during workup, more or less extensive hydrolysis leading to **10** can occur.

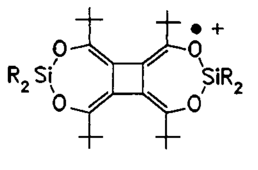


$$3 + H_2O \longrightarrow [Ph\text{–}\overset{+}{I}\text{–}O\text{–}\underset{OH}{I}\text{–}Ph] \cdot BF_4^- + HBF_4$$

10

Thus, samples of **3** always contain a certain amount of **10**, that is considered to be the hydrogen source in the reaction leading to **9**. The exact mode of formation of **9** as well as the influence of the solvent[12] remain a matter of speculation, however.

The experiments described above have documented the failure to convert **1** into tetrapivaloylcyclobutadiene by oxidative desilylation of **1**. In principle, electrochemical oxidation is another way to transform a [4]radialene into a cyclobutadiene. So far, the fully reversible, four-step one-electron electrochemical oxidation of an electron-rich [4]radialene to a cyclobutadiene bearing four cationic substituents has been realized only in one case[22]; for tetra(1,3-dithiol-2-ylidene)cyclobutane, on the other hand, only the first two out of four one-electron oxidation steps were reversible[23]. Successive removal of two electrons from **1** should lead to radical cation **11** and dication **12**; the formation of the cyclobutadiene **13** bearing four cationic substituents would require a four-electron oxidation of **1**. In all these cationic species, the positive charges are stabilized both by the oxygen and *tert*-butyl substituents. According to preliminary results[24], the cyclic voltammogram of **1** (solvent CH_2Cl_2, supporting electrolyte Bu_4NClO_4) reveals a complicated, partly irreversible redox behavior that does not allow a straightforward explanation. A meaningful discussion must await further investigations.



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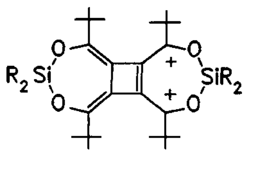

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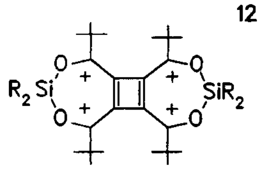

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Crystal Structure Analysis of 9

A plot of the molecular structure in the crystal is shown in Figure 1, selected bond distances and angles are given in Table 1. To the best of our knowledge, **9** represents the first fluorosilyl enol ether whose crystal structure has been determined. When we focus on this structural moiety, the bond angle C4–O3–Si2 (145.4°) is worth mentioning, since it is larger than in other silyl enol ethers investigated so far (127.7–137.4°[14]) including a siloxy-substituted carbene complex[15] (143.8°). The widening of this angle may be attributed to the reduced electronegativity of the silyl group caused by fluoro substitution[16].

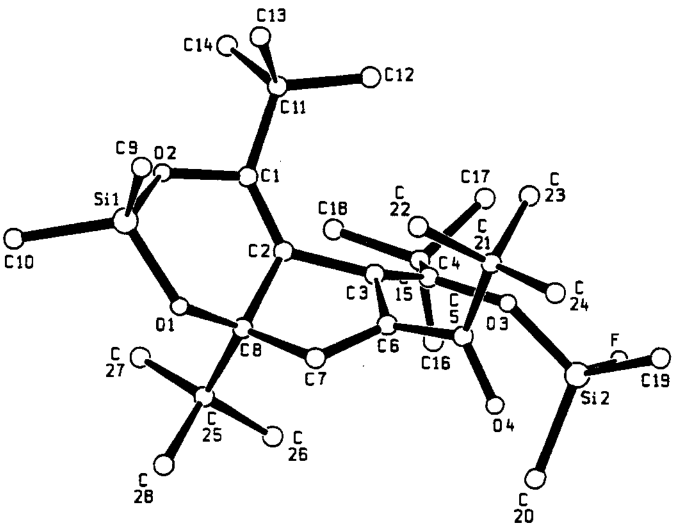

Figure 1. SCHAKAL[13] plot of **9**

Table 1. Selected bond distances [Å] and angles [deg] in **9**. Estimated standard deviations are in parentheses

Si1 - O1	1.625(4)	O3 - C4	1.384(6)
Si1 - O2	1.629(4)	O4 - C5	1.212(6)
Si1 - C9	1.823(7)	C1 - C2	1.316(7)
Si1 - C10	1.826(6)	C2 - C3	1.510(7)
Si2 - F	1.600(4)	C2 - C8	1.562(7)
Si2 - O3	1.612(4)	C3 - C4	1.343(7)
Si2 - C19	1.832(8)	C3 - C6	1.491(7)
Si2 - C20	1.786(9)	C5 - C6	1.479(8)
O1 - C8	1.447(5)	C6 - C7	1.321(7)
O2 - C1	1.401(6)	C7 - C8	1.507(7)

O1 - Si1 - O2	102.9(2)	C3 - C2 - C8	104.4(4)
O1 - Si1 - C9	106.8(3)	C2 - C3 - C4	133.5(5)
O1 - Si1 - C10	113.1(3)	C2 - C3 - C6	101.6(4)
O2 - Si1 - C9	115.1(3)	C4 - C3 - C6	121.9(5)
O2 - Si1 - C10	108.4(3)	O3 - C4 - C3	114.7(5)
C9 - Si1 - C10	110.4(3)	O3 - C4 - C15	112.3(4)
F - Si2 - O3	107.1(2)	C3 - C4 - C15	133.0(5)
F - Si2 - C19	104.1(3)	O4 - C5 - C6	120.2(6)
F - Si2 - C20	107.8(3)	O4 - C5 - C21	121.7(5)
O3 - Si2 - C19	109.2(3)	C6 - C5 - C21	117.7(5)
O3 - Si2 - C20	113.3(3)	C3 - C6 - C5	129.6(5)
C19 - Si2 - C20	114.6(4)	C3 - C6 - C7	109.6(5)
Si1 - O1 - C8	124.0(3)	C5 - C6 - C7	120.8(5)
Si1 - O2 - C1	120.7(3)	C6 - C7 - C8	113.5(5)
Si2 - O3 - C4	145.4(4)	O1 - C8 - C2	113.4(4)
O2 - C1 - C2	117.9(5)	O1 - C8 - C7	104.8(4)
O2 - C1 - C11	107.3(4)	O1 - C8 - C25	106.6(4)
C2 - C1 - C11	134.7(5)	C2 - C8 - C7	98.8(4)
C1 - C2 - C3	130.1(5)	C2 - C8 - C25	118.1(4)
C1 - C2 - C8	122.0(4)	C7 - C8 - C25	114.5(4)

The geometry around Si2 points to an incipient pentacoordination at this atom, the tetrahedral geometry being weakly distorted towards a trigonal bipyramid in which F and O4 occupy the apical positions. In agreement with this structural change, the angle O4···Si2–F is 177.9°, the three angles between F and the "equatorial" substituents (O3, C19, C20) are smaller (107.1, 104.1, 107.8°) and two out of the three angles between the "equatorial" substituents (109.2, 113.3, 114.6°) are larger than the tetraedral angle of 109.5°. However, the contact distance between O4 and Si2 [3.350(4) Å] is not distinctly smaller than the sum of the van der Waals radii of silicon and oxygen (3.35[17]–3.62[18]); moreover, it is much larger than in those pentacoordinate organosilicon compounds in which the trigonal-bipyramidal geometry is more closely approximated or fully established[19]. Even the intermolecular (C=)O···Si contact in silyl acetate is as low as 2.721 Å[20]. Since Si2 maintains a van der Waals contact not only to O4 but also to C5 (C5···Si2 3.525 Å, Si2···C5–O4 71.8°, Si2···O4–C5 88.2°), the geometry around Si2 may be the result of an interaction between the silyl group and the carbonyl π system. A tighter pentacoordination at Si2 in **9** would close a seven-membered ring, but obviously, the attractive Si···O interaction could not compensate the additonal ring strain. This is in agreement with investigations on compounds of type Ar–COO–$[CH_2]_n$–SiX_3 (X = Cl, F), for which the ability to undergo intramolecular Si ← O coordination is lost gradually from $n = 1$ to $n = 3$[21].

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Experimental

IR: Perkin-Elmer IR 397. – ^{1}H NMR: Varian EM 390 (90 MHz), solvent $CDCl_3$, $CHCl_3$ as internal standard [δ($CHCl_3$) = 7.24]. – ^{13}C NMR: Bruker AM 400 (100.6 MHz), solvent $CDCl_3$; solvent signal used as internal standard [δ($CDCl_3$) = 77.0). – MS: Finnigan MAT 90. – X-ray diffraction: Enraf Nonius CAD 4. – Melting points: Copper block, temperatures given are not calibrated. – Column chromatography: silica gel, particle size 0.063–0.2 mm. – Elemental analyses: Perkin-Elmer EA 2400. – Solvents were dried by standard procedures.

1,5-Bis(1,1-dimethylethyl)-7,8-bis(2,2-dimethyl-1-oxopropyl)cyclobuta[e][1,3,2]dioxasilepine (**2**): A solution of 4-methyl-3*H*-1,2,4-triazole-3,5(4*H*)-dione[7] (0.099 g, 0.88 mmol) in toluene (3 ml) is added to a solution of **1**[2] (0.210 g, 0.41 mmol) in toluene (10 ml), and the mixture is refluxed until the red color has dissappeared (ca. 3 h). The solvent is evaporated at 0.01 Torr, pentane (10 ml) is added to the residue, and undissolved material is removed by filtration. Crystallization yields 0.110 g (60%) of **2**, m.p. 152°C. – IR (KBr): ṽ = 1680 cm^{-1} (C=O), 1622, 1519, 1484, 1468, 1292, 1270, 1208, 1085, 900, 820. – ^{1}H NMR (90 MHz): δ = 0.27 (s, 6H, $SiMe_2$), 1.07 (s, 18H, CMe_3), 1.17 (s, 18H, CMe_3). – ^{13}C NMR: δ = –4.2 ($SiMe_2$), 26.9 (C*Me*$_3$), 29.0 (C*Me*$_3$), 37.4 (CMe_3), 44.6 (CMe_3), 119.1 (*C*=C–O), 141.2 (C=C), 146.7 (C=*C*–O), 211.1 (C=O). – MS (70 eV, EI), *m/z* (%): 446 (12) [M$^+$], 389 (82), 361 (22), 332 (24), 305 (34), 57 (100).

$C_{26}H_{42}O_4Si$ (446.6) Calcd. C 69.93 H 9.47
Found C 69.4 H 9.25

1,1′,1″,1‴-[(3α,4α)-1-Cyclobutene-1,2,3,4-tetrayl]tetrakis(2,2-dimethyl-1-propanone) [(*Z*)-**4**]: A mixture of radialene **1**[2] (0.433 g, 0.85 mmol) and salt **3**[11] (0.500 g, 0.85 mmol) in dichloromethane (10 ml) is stirred for 12 h. The solvent is replaced by ether (15 ml), the solution washed with water (10 ml) and dried (Na_2SO_4). The residue obtained after evaporation of the solvent is washed with pentane; yield of (*Z*)-**4**: 0.180 g (55%), m.p. 210°C (dec.). – IR (KBr): ṽ = 1682/1670 cm^{-1} (C=O); 1610, 1455, 1078. – ^{1}H NMR (90 MHz): δ = 1.07 (s, 18H, CMe_3), 1.10 (s, 18H, CMe_3), 4.52 (s, 2H). – ^{13}C NMR: δ = 26.5 (C*Me*$_3$), 26.7 (C*Me*$_3$), 43.8 (CMe_3), 44.4 (CMe_3), 52.5 [*J*(C,H) = 140 Hz, CH ring], 144.6 (C=C), 205.9 (C=O), 210.2 (C=O). – Although pure as determined by NMR spectroscopy, the product has not given a satisfactory microanalysis. Recrystallization did not improve the purity. Further characterization is possible by epimerization to (*E*)-**4** with dilute aqueous NaOH.

1,1′,1″,1‴-[(3α,4β)-1-Cyclobutene-1,2,3,4-tetrayl]tetrakis(2,2-dimethyl-1-propanone) [(*E*)-**4**]: A mixture of radialene **1**[2] (0.900 g, 1.78 mmol) and salt **3**[11] (2.07 g, 3.6 mmol) in dichloromethane (10 ml) is stirred for 12 h. Workup by column chromatography [silica gel, 30 g, eluant ether/petroleum ether, 1:1 (v/v)] yields 0.230 g (33%) of (*E*)-**4**, m.p. 145°C. – IR (KBr): ṽ = 1685/1668 cm^{-1} (C=O), 1605, 1472, 1455, 1052. – ^{1}H NMR (90 MHz): δ = 1.14 (s, 18H, CMe_3), 1.18 (s, 18H, CMe_3), 4.30 (s, 2H). – ^{13}C NMR: δ = 26.25 (C*Me*$_3$), 26.29 (C*Me*$_3$), 43.7 (CMe_3), 44.5 (CMe_3), 51.2 [*J*(C,H) = 146 Hz, CH ring], 145.6 (C=C), 204.8 (C=O), 211.3 (C=O).

$C_{24}H_{38}O_4$ (390.6) Calcd. C 73.81 H 9.80
Found C 73.8 H 9.70

1,5-Bis(1,1-dimethylethyl)-8-(2,2-dimethyl-1-oxopropyl)-7-[(Z)-(fluorodimethylsilyloxy)-2,2-dimethylpropylidene]-3,3-dimethyl-2,4-dioxa-3-silabicyclo[4.3.0]nona-5,8-diene (**9**): A mixture of **1**[2] (0.197 g, 0.35 mmol) and salt **3**[11] (0.430 g, 0.77 mmol) in tetrachloromethane (10 ml) is stirred for 5 h. After evaporation of the solvent, the product is extracted from the residue into ether (2 × 10 ml) and recrystallized from ether/pentane; yield of **9**: 0.138 g (75%), m.p. 189°C. – IR (KBr): ṽ = 1675 cm^{-1} (C=O), 1592, 1568, 1550, 1470, 1455, 1390, 1358, 1255, 1157, 1135. – ^{1}H NMR (90 MHz): δ = 0.18 (s, 3H, 3-SiMe), 0.34 (d, *J* = 2 Hz, 3H, SiMeF), 0.40 (s, broad, 6H, 3-SiMe + SiMeF), 1.14, 1.27, 1.30, 1.37 (each s, each 9H, CMe_3), 5.99 (s, 1H, =CH). – ^{13}C NMR: δ = –0.75 [d, *J*(C,F) = 25.8 Hz, SiMeF], –0.48 [d, *J*(C,F) = 28.4, SiMeF], 0.45 (3-SiMe), 1.8 (3-SiMe); 26.9, 27.7, 28.9, 29.9 (C*Me*$_3$); 38.0, 39.2, 40.6,

Table 2. Positional parameters and equivalent isotropic displacement factors for **9** in the crystal. Estimated standard deviations are in parentheses

Atom	*x/a*	*y/b*	*z/c*	B_{eq}
Si1	1.0015(2)	0.12033(6)	0.2923(1)	3.79(4)
Si2	1.5251(2)	0.10718(8)	0.9360(1)	5.38(5)
F	1.6325(4)	0.1480(2)	1.0061(3)	8.2(1)
O1	1.1167(4)	0.0817(1)	0.3687(3)	3.31(9)
O2	1.0593(4)	0.1718(1)	0.3531(3)	3.46(9)
O3	1.4032(4)	0.1343(1)	0.8366(3)	3.80(9)
O4	1.2940(4)	0.0214(2)	0.7969(3)	5.2(1)
C1	1.1129(6)	0.1750(2)	0.4715(4)	3.0(1)
C2	1.2119(5)	0.1429(2)	0.5231(4)	2.6(1)
C3	1.2752(5)	0.1332(2)	0.6483(4)	2.8(1)
C4	1.3764(6)	0.1559(2)	0.7309(4)	2.9(1)
C5	1.2101(6)	0.0533(2)	0.7517(4)	3.5(1)
C6	1.2403(5)	0.0808(2)	0.6559(4)	2.9(1)
C7	1.2300(6)	0.0598(2)	0.5568(4)	3.0(1)
C8	1.2392(6)	0.0948(2)	0.4641(4)	2.9(1)
C9	0.8149(7)	0.1023(3)	0.2984(5)	5.9(2)
C10	1.0131(7)	0.1231(3)	0.1451(5)	5.3(2)
C11	1.0291(6)	0.2165(2)	0.5110(4)	3.6(1)
C12	1.0656(7)	0.2239(2)	0.6389(5)	5.2(2)
C13	0.8637(7)	0.2059(3)	0.4741(6)	6.2(2)
C14	1.0564(8)	0.2629(2)	0.4512(5)	5.7(2)
C15	1.4718(6)	0.2004(2)	0.7320(4)	3.7(1)
C16	1.6364(6)	0.1857(2)	0.7607(5)	5.1(2)
C17	1.4552(8)	0.2360(2)	0.8232(5)	5.6(2)
C18	1.4416(7)	0.2255(2)	0.6180(5)	5.2(2)
C19	1.4326(8)	0.0817(3)	1.0387(5)	7.4(2)
C20	1.6377(8)	0.0652(3)	0.8837(7)	7.5(2)
C21	1.0625(7)	0.0620(2)	0.7800(4)	3.7(1)
C22	0.9372(7)	0.0607(3)	0.6721(6)	7.6(2)
C23	1.0633(7)	0.1107(3)	0.8377(5)	5.5(2)
C24	1.0374(8)	0.0228(3)	0.8593(6)	7.3(2)
C25	1.3842(6)	0.0905(2)	0.4226(4)	3.1(1)
C26	1.5198(6)	0.0930(2)	0.5252(5)	4.5(2)
C27	1.3920(7)	0.1316(2)	0.3430(5)	4.6(2)
C28	1.3883(7)	0.0426(2)	0.3629(5)	4.9(2)

44.5 (CMe$_3$); 91.5 (C-1), 121.7, 126.8, 138.8 [J(C,H) = 171 Hz, C-9], 148.7, 148.9, 150.6, 208.3 (C=O). – MS (70 eV, EI), m/z (%): 524 (2) [M$^+$], 468 (76), 467 (91), 411 (100), 410 (75), 395 (83), 217 (77), 77 (22), 57 (44).

$C_{28}H_{49}FO_4Si_2$ (524.9) Calcd. C 64.07 H 9.41
Found C 64.3 H 9.3

X-Ray Crystal Structure Analysis of **9**: *Crystal data:* $C_{28}H_{49}FO_4Si_2$, molecular mass 524.9, monoclinic, space group $P2_1/c$, a = 9.290(4), b = 27.641(11), c = 12.187(4) Å, β = 104.98(3)°, Z = 4, d_{calc} = 1.15 g cm^{-3}. – *Data collection:* Crystal size 0.6 × 0.3 × 0.3 mm, monochromatized Mo-K_α radiation, 3998 reflections measured ($2.0 \le \Theta \le 24.0°$), 3686 unique, 2469 observed [$I > 2\sigma(I)$], $\omega/2\Theta$ scan, scan width $(1.30 + 0.35 \tan\Theta)°$. The intensity loss (up to 4.5%) during data collection was corrected anisotropically. – *Structure solution and refinement*[25]: Structure solution by direct methods (MULTAN), refinement by a full-matrix least-squares method. Hydrogen atoms were either located in a difference Fourier map or calculated; they were included in the structure-factor calculation with B = 7.0 or 8.0 Å^2. Refinement converged at R = 0.069, R_w = 0.074, $w = 4 \cdot F^2_{obs}/[\sigma(I)^2 + (0.04 \cdot F^2_{obs})^2]$; shift/error ratio ≤ 0.02. Positional and thermal parameters are given in Table 2[26].

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[26] Further crystal structure data have been deposited at Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-7514 Eggenstein-Leopoldshafen 2, Germany, Inquiries should be accompanied by the depository number CSD-56522, the names of the authors and the reference to this publication.

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