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Tungsten-catalyzed reactions of silacyclopropenes

Joji Ohshita and Mitsuo Ishikawa *

Department of Applied Chemistry, Faculty of Engineering, Hiroshima University, Higashi-Hiroshima 724 (Japan)

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Abstract

Hexacarbonyltungsten(0)-catalyzed reactions of silacyclopropenes are reported. Treatment of 1-mesityl-1-methyl-3-phenyl-2-(trimethylsilyl)-1-silacyclopropene and 1-methyl-3-phenyl-1,2-bis(trimethylsilyl)-1-silacyclopropene with a catalytic amount of $W(CO)_6$ afforded phenyl(trimethylsilyl)acetylene and the respective silylenes which could be readily trapped by 2,3-dimethyl-1,3-butadiene. Mesitylmethylsilylene could also be trapped by diphenylacetylene. When 1-mesityl-3-phenyl-1,2-bis(trimethylsilyl)-1-silacyclopropene was heated with a catalytic amount of $W(CO)_6$ at 190 °C, a novel isomerization was observed.

Introduction

Considerable interest has been focussed on the chemistry of silicon-containing small ring compounds [1]. To date, many studies on the transition-metal-catalyzed reactions of silacyclopropenes have been reported [2–8].

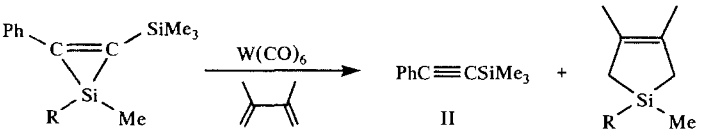
Recently, we have found that the nickel(0)- [4–7] and palladium(0)-catalyzed reactions [8] of silacyclopropenes produce reactive intermediates such as metalasilacyclobutenes arising from insertion of metals into the Si–C bond in the silacyclopropene ring and silapropadiene-metal complexes formed from isomerization of the resulting metalasilacyclobutenes. In an effort to learn much more about the chemical behaviour of the silacyclopropenes towards the transition metal complexes, we have investigated the reaction of the silacyclopropene with transition metal complexes other than those of nickel and palladium. In this paper, we wish to report the tungsten(0)-catalyzed reaction of silacyclopropenes which proceed in a quite different fashion from the nickel- and palladium-catalyzed reactions. The reaction involves abstraction of silylene from silacyclopropene by the transition metal complex.

Results and discussion

Hexacarbonyltungsten-catalyzed generation of silylene from silacyclopropenes

When a THF solution of 1-mesityl-1-methyl-3-phenyl-2-trimethylsilyl-1-silacyclopropene (Ia) was heated to 120 °C for 20 h in a sealed glass tube with slight excess of 2,3-dimethyl-1,3-butadiene in the presence of a catalytic amount of $W(CO)_6$, phenyl(trimethylsilyl)acetylene (II) and 1-mesityl-1,3,4-trimethyl-1-silacyclopent-3-ene (IIIa) were obtained in 38 and 40% yields, respectively, in addition to an unidentified product (less than 5% yield) whose mass spectrum showed a parent peak at 418, corresponding to a 1 : 1 adduct of Ia and 2,3-dimethyl-1,3-butadiene. In the absence of the tungsten catalyst under the same conditions, the starting silacyclopropene Ia was recovered unchanged. These results clearly show that the reaction involves the tungsten-catalyzed abstraction of mesitylmethylsilylene from the silacyclopropene Ia. The silylene thus formed can be readily trapped by 2,3-dimethyl-1,3-butadiene, which is known as an efficient silylene trapping agent [9], to give IIIa.

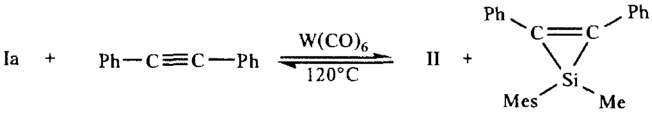
Similarly, the reaction of 1-methyl-3-phenyl-1,2-bis(trimethylsilyl)-1-silacyclopropene (Ib) in the presence of 2,3-dimethyl-1,3-butadiene under the same conditions afforded II and 1,3,4-trimethyl-1-trimethylsilyl-1-silacyclopent-3-ene (IIIb) in 22 and 25% yields, respectively, indicating the formation of methyl(trimethylsilyl)silylene.



Ia, R = Mes
Ib, R = SiMe3

IIIa, R = Mes
IIIb, R = SiMe3

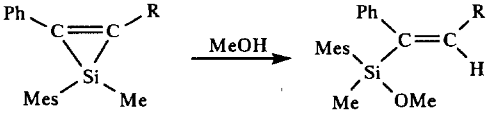
Mesitylmethylsilylene can also be trapped by diphenylacetylene. Thus, a mixture of Ia and diphenylacetylene in a THF solution was heated to 120 ° C with a catalytic amount of $W(CO)_6$, and the progress of the reaction was monitored by GLC. After 32 h, GLC analysis of the reaction mixture showed the formation of II and 1-mesityl-1-methyl-2,3-diphenyl-1-silacyclopropene (IV), together with the starting compound Ia. The ratio of IV to Ia was determined to be 1 : 3. This ratio increased with increasing the reaction time, and after 220 h, it reached a constant value of 2 : 3.



IV

In sharp contrast to the nickel- and palladium-catalyzed reaction of silacyclopropenes in the presence of acetylenes, in which adducts arising from the reaction of silacyclopropenes with acetylenes are always produced [2–7], the present reaction afforded no adduct derived from the reaction of diphenylacetylene and Ia.

Compound IV is extremely air-sensitive, and therefore, all attempts to isolate IV were unsuccessful. However, its GC-MS spectrum shows a parent ion peak at *m/e* 340, corresponding to the calculated molecular weight of the product arising from the reaction of mesitylmethylsilylene with diphenylacetylene. Moreover, the structure of IV could be verified by an indirect method. Thus, treatment of the reaction mixture with a large excess of methanol led to the formation of methanol adduct (V) in quantitative yield, along with methoxysilane (VI) produced from the reaction of Ia with methanol.



IV, R = Ph
Ia, R = $SiMe_3$

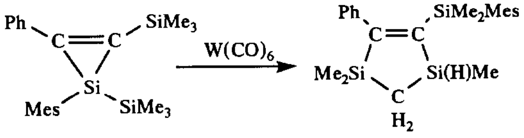
V, R = Ph
VI, R = $SiMe_3$

Adducts V and VI were identified as (*E*)-1-[mesityl(methoxy)methylsilyl]-1,2-diphenylethene and (*E*)-1-[mesityl(methoxy)methylsilyl]-1-phenyl-2-trimethylsilylethene, respectively, by spectroscopic analysis, as well as by elemental analysis (see Experimental Section).

Isomerization of silacyclopropene Ic

Treatment of 1-mesityl-3-phenyl-1,2-bis(trimethylsilyl)-1-silacyclopropene (Ic) in the presence of the tungsten catalyst at 120 °C gave no volatile product. The starting compound Ic was recovered unchanged. Similar treatment of Ic at 190 °C in the presence of 2,3-dimethyl-1,3-butadiene, however, gave II in quantitative yield. In the reaction, mesityl(trimethylsilyl)silylene should be formed, but nevertheless no silylene adduct, analogous to IIIa and IIIb, was detected by either GLC or spectrometric analysis. The silacyclopentene was probably formed, but this product would undergo ring-opening polymerization to give nonvolatile substances.

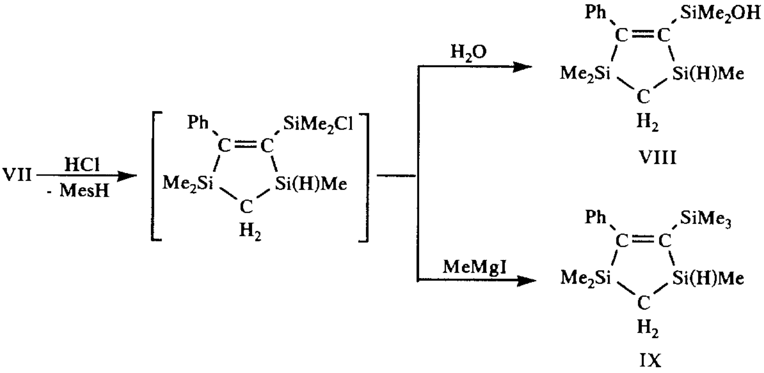
To our surprise, when a similar reaction was carried out in the absence of 2,3-dimethyl-1,3-butadiene at 190 °C for 20 h in a sealed glass tube, the isomerization product, 4-(mesityldimethylsilyl)-5-phenyl-1,1,3-trimethyl-1,3-disilacyclopent-4-ene (VII) was obtained in 33% yield, together with six minor products (ca. 5% each). Mass spectra of all minor products showed a parent peak at *m/e* 394 which is identical with that of Ic. In this reaction, no phenyl(trimethylsilyl)acetylene was detected, indicating that no silylene species was produced. The real catalyst produced in this system presumably differs from that formed in the presence of 2,3-dimethyl-1,3-butadiene.



Ic

VII

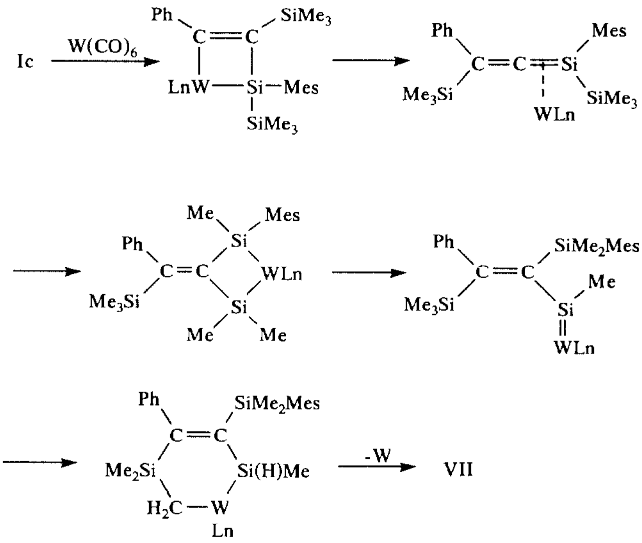
The structure of VII was verified by spectrometric analysis, elemental analysis, and also by chemical reactions. The IR spectrum of VII shows a strong absorption band at 2104 cm^{-1}, due to the stretching frequency of a Si–H bond. The ^1H NMR spectrum shows four singlets and one doublet signal at δ 0.11, 0.14, 0.41, 0.47, and 0.30 ppm, due to four MeSi and one MeSi (H) protons, as well as multiplet signal at δ 4.54 ppm attributed to hydrogen on the ring silicon. Two doublets of doublet at δ -2.84 and 0.09 ppm can be assigned as methylene protons in the silacyclopentene ring. The ^{13}C NMR spectrum shows six signals due to six different kinds of silyl-substituted sp^3 carbons. That the carbon atom appeared in the highest field



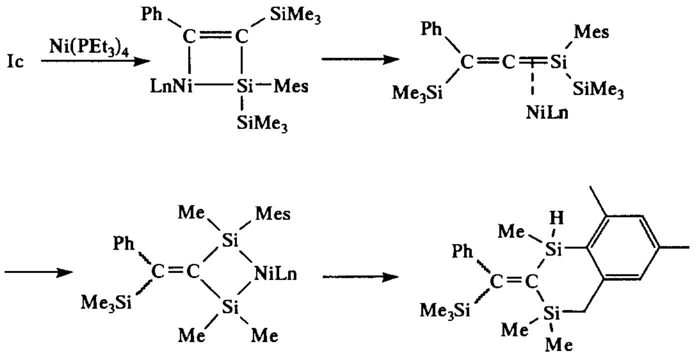
Scheme 1

may be assigned to the carbon having two silicon atoms. These results are wholly consistent with the proposed structure.

In order to get more information about the structure of VII, we investigated its chemical behaviour (Scheme 1). Treatment of a benzene solution of VII with dry hydrogen chloride gas, followed by hydrolysis of the resulting product afforded 4-(hydroxydimethylsilyl)-1,1,3-trimethyl-5-phenyl-1,3-disilacyclopent-4-ene (VIII) in 51% yield. All spectral data obtained for VIII reveal the absence of a mesityl group, but the presence of a hydroxy group. Next, we carried out the methylation reaction of the product obtained from the reaction of VII with hydrogen chloride gas. If the structure of VII is correct, two non-equivalent methyl groups on the silicon atom bearing a mesityl group should be transformed into equivalent by replacing of this mesityl group with a methyl group. When VII was treated with dry hydrogen chloride gas, followed by methylation of the resulting chlorosilane with methyl



Scheme 2



Xa, b

Scheme 3

magnesium iodide, compound IX was obtained in 89% yield. As expected, ^{1}H and ^{13}C NMR spectra of the product IX show the presence of three non-equivalent silyl methyl groups and one trimethylsilyl group in the molecule.

A possible mechanistic interpretation for the formation of VII is presented in Scheme 2. Tungstenasilacyclobutene arising from insertion of a tungsten species into an Si–C bond of the starting compound Ic rearranges to a silapropadiene-tungsten complex intermediate via a 1,2-shift of a trimethylsilyl group. The silapropadiene-tungsten complex thus formed isomerizes to a tungstenadisilacyclobutane, in which migration of a mesityl group on the ring silicon atom to another ring silicon is involved, to give a silylene-complex. Finally, the resulting silylene-complex undergoes intramolecular insertion into a C–H bond of a methyl group of a trimethylsilyl moiety to afford the five-membered cyclic compound VII. As reported previously [7], the nickel-catalyzed reaction of Ic produced disilacyclohexane derivatives (Xa and Xb). This reaction presumably proceeds with formation of a transient silapropadiene-nickel complex which rearranges to a nickeladisilacyclobutane intermediate (Scheme 3). It seems likely that the present reaction proceeds in a similar manner to that of the nickel-catalyzed isomerization of Ic. In the case of the nickeladisilacyclobutane, however, activation of the C–H bond of a mesityl methyl group occurs, while the tungstenadisilacyclobutane would involve the formation of the silylene-complex.

Experimental

General

All reactions were carried out under an atmosphere of purified argon. ^{1}H and ^{13}C NMR spectra were determined with a JEOL Model JNM-GX-500 spectrometer, a JEOL Model JNM-FX-90A spectrometer, and a JEOL Model JNM-PMX-60A spectrometer using deuteriochloroform and carbon tetrachloride solution containing tetramethylsilane as an internal standard. Infrared spectra were recorded on a Perkin-Elmer 1600 FT-infrared spectrometer. Mass spectra were measured on a Shimazu Model GCMS-QP 1000 and a JEOL Model JMS-D300 equipped with JMA-2000 data processing system.

Materials

All starting phenylethynylpolysilanes were prepared as reported in the literature [4,8,11]. Hexane and THF used as solvents were dried over lithium aluminum hydride and distilled just before use.

Synthesis of silacyclopropenes (Ia,Ib, and Ic)

All silacyclopropenes Ia [10], Ib [4], and Ic [8] were synthesized as reported in the literature. In a 25 mL reaction vessel fitted with a 6-W low pressure mercury lamp was placed a solution of phenylethynylpolysilane in 25 mL of hexane. The solution was irradiated until 90–95% of phenylethynylpolysilane was consumed. After evaporation of the solvent, the residue was distilled under reduced pressure to give silacyclopropene, Ia (60–65% yield), Ib (40–45% yield), and Ic (75–80% yield). For Ia: MS *m/e* 336 (M^+). ^{1}H NMR (THF, 60 MHz: δ 0.26 (s, 9H, Me_3Si); 0.49 (s, 3H, MeSi); 2.16 (s, 3H, *p*-Me); 2.58 (s, 6H, *o*-Me); 6.60 (br s, 2H, mesityl ring protons); 6.93–7.43 (m, 5H, Ph). ^{13}C NMR (THF/C_6D_6 = 2/1, 22.5 MHz): δ −2.4, 0.4, 21.0, 24.4, 127.8, 128.1, 128.5, 128.9, 132.9, 137.3, 139.1, 143.2, 160.9, 177.6. For Ib: MS *m/e* 290 (M^+). ^{1}H NMR (THF): δ 0.02 (s, 3H, MeSi); 0.15 (s, 9H, Me_3Si); 0.26 (s, 9H, Me_3Si); 7.00–7.47 (m, 5H, Ph). For Ic: MS *m/e* 394 (M^+). ^{1}H NMR (THF): δ 0.16 (s, 9H, Me_3Si); 0.29 (s, 9H, Me_3Si); 2.16 (s, 3H, *p*-Me); 2.53 (s, 6H, *o*-Me); 6.60 (br s, 2H, mesityl ring protons); 6.90–7.50 (m, 5H, Ph).

Reaction of Ia in the presence of 2,3-dimethyl-1,3-butadiene

A mixture of 0.700 mmol of Ia, 0.5 mL of 2,3-dimethyl-1,3-butadiene, 18 mg (10 mol%) of $W(CO)_6$, and 0.0767 mmol of nonadecane as an internal standard in THF was heated at 120 °C for 20 h in a sealed glass tube. GLC analysis of the resulting mixture showed the presence of II (33%), IIIa (35%), and Ia (13%). The solvent was distilled off and the residue was treated with MPLC (silica gel, eluent hexane) to give pure II and IIIa. All spectral data obtained for II were identical with those of an authentic sample prepared from the reaction of phenylethynyl lithium and chlorotrimethylsilane [12]: MS *m/e* 174 (M^+). IR ν(C≡C) 2159 cm^{-1}; ^{1}H NMR (CCl_4, 100 MHz): δ 0.27 (s, 9H, Me_3Si); 7.08–7.38 (m, 5H, Ph). ^{13}C NMR (C_6D_6, 22.5 MHz): δ −0.1, 94.2, 106.0, 123.9, 128.4, 128.9, 132.2. For IIIa: MS *m/e* 244 (M^+). ^{1}H NMR ($CDCl_3$, 90 MHz): δ 0.24 (s, 3H, MeSi); 1.60 (br s, 2H, silacyclopentene ring protons); 1.74 (br s, 6H, Me–C=C); 1.82 (br s, 2H, silacyclopentene ring protons); 2.26 (s, 3H, *p*-Me); 2.38 (s, 6H, *o*-Me); 6.83 (br s, 2H, mesityl ring protons). ^{13}C NMR ($CDCl_3$, 22.5 MHz): δ −2.0, 21.0, 24.0, 27.0, 28.6, 128.3, 130.0, 132.7, 138.7, 143.9. Anal. Found: C, 78.58; H, 9.86. $C_{16}H_{24}Si$ calcd.: C, 78.62; H, 9.90%.

Reaction of Ib in the presence of 2,3-dimethyl-1,3-butadiene

A mixture of 0.375 mmol of Ib, 0.5 mL of 2,3-dimethyl-1,3-butadiene, 13 mg (10 mol%) of $W(CO)_6$, and 0.123 mmol of tetradecane as an internal standard in THF was heated at 120 °C for 20 h in a sealed glass tube. The resulting mixture was analyzed by GLC as being II (22% yield) and IIIb (25% yield). At this stage ca. 90% of starting Ib was consumed. Product II and IIIb were isolated by preparative GLC. All spectral data obtained for II were identical with those of the authentic sample. For IIIb: MS *m/e* 198 (M^+). ^{1}H NMR (C_6D_6, 90 MHz); δ 0.06 (s 9H, Me_3Si); 0.11 (s, 3H, MeSi); 1.31 (br d, 2H, J = 11.7 Hz, silacyclopentene ring protons); 1.48 (br d, 2H, $J = 11.7$ Hz, silacyclopentene ring protons); 1.67 (br s, 6H, Me–C); ^{13}C NMR (C_6D_6, 22.5 MHz): δ −4.2, −1.9, 19.4, 24.5, 130.9. Anal. Found: C, 60.30; H, 11.04. $C_{10}H_{22}Si_2$ calcd.: C, 60.52; H, 11.17%.

Reaction of Ia with diphenylacetylene

A mixture of 0.382 mmol of Ia, 68 mg (0.382 mmol) of diphenylacetylene, and 0.174 mmol of nonadecane in THF was heated to 120 ° C for 20 h with 10 mol% of $W(CO)_6$ in a sealed glass tube. The reaction mixture was analyzed by GLC and GC-MS as being Ia (32% yield), II (17% yield), and IVa (13% yield; MS m/e 340 (M^+)). The mixture was treated with 0.2 mL of methanol at room temperature. GLC analysis of the resulting mixture showed that compound IV and Ia were completely converted to relative V and VI. Compounds V and VI were isolated by preparative GLC. For V: MS m/e 414 (M^+). IR ν(Si–O) 1085 cm^{-1}. ^1H NMR (CCl_4, 60 MHz): δ 0.49 (s, 3H, MeSi); 2.22 (s, 3H, p-Me); 2.35 (s, 6H, o-Me); 3.40 (s, 3H, MeO); 6.26–7.20 (m, 12H, ring protons); 7.46 (s, 1H, olefinic proton). ^{13}C NMR ($CDCl_3$, 22.5 MHz): δ −2.0, 21.0, 23.9, 50.4, 125.9, 127.2, 127.4, 127.9, 128.0, 128.2, 128.5, 128.8, 129.1, 129.7, 139.3, 139.6, 144.1, 145.2. Anal. Found: C, 80.37; H, 7.47. $C_{25}H_{28}OSi$ calcd.: C, 80.59; H, 7.57%. For VI: MS m/e 368 (M^+). IR ν(Si–O) 1086 cm^{-1}. ^1H NMR (CCl_4, 60 MHz): δ 0.18 (s, 9H, Me_3Si); 0.42 (s, 3H, Me–Si); 2.21 (s, 3H, p-Me); 2.29 (s, 6H, o-MeO); 3.40 (s, 3H, MeO); 6.35 (s, 1H, olefinic proton); 6.63 (br s, 2H, mesityl ring protons); 6.70–7.22 (m, 5H, Ph). ^{13}C NMR ($CDCl_3$, 22.5 MHz): δ −0.8, 20.9, 23.9, 50.3, 125.9, 127.6 (2 carbons), 127.8, 127.9, 129.0, 129.3, 139.1, 145.1, 146.6. Anal. Found: C, 71.67; H, 8.71. $C_{22}H_{32}OSi_2$ calcd.: C, 71.68; H, 8.75%.

Reaction of Ic in the presence of 2,3-dimethyl-1,3-butadiene at 120 ° C

A mixture of 0.568 mmol of Ic, 0.5 mL of 2,3-dimethyl-1,3-butadiene, 24 mg (10 mol%) of $W(CO)_6$, and 0.0719 mmol of eicosane as an internal standard in 1 mL of THF was heated at 120 ° C for 20 h in a sealed glass tube. GLC and the mass spectrometric analysis of the resulting mixture showed that all Ic remained unchanged.

Reaction of Ic in the presence of 2,3-dimethyl-1,3-butadiene at 190 ° C

A mixture of 0.577 mmol of Ic, 0.5 mL of 2,3-dimethyl-1,3-butadiene, 14 mg (7 mol%) of $W(CO)_6$, and 0.259 mmol of eicosane as an internal standard in 1 mL of THF was heated at 190 ° C for 20 h in a sealed glass tube. The resulting mixture was analyzed by GLC as being II (100% yield). The retention time of GLC and the mass spectrum of II was identical with those of the authentic sample.

Isomerization reaction of Ic

A mixture of 0.427 mmol of Ic, 15 mg (10 mol%) of $W(CO)_6$, and 0.155 mmol of docosane as an internal standard was heated at 190 ° C in a sealed glass tube. Analysis of the resulting mixture by GLC showed the formation of VII (33% yield) together with six unidentified products (less than 5% yields). Pure VII was separated from the mixture by MPLC (silica gel, eluent hexane): MS m/e 394 (M^+). IR ν(Si–H) 2104 cm^{-1}. ^1H NMR (C_6D_6, 500 MHz): δ −2.84 (dd, 1H, $J = 14.3$, 2.5 Hz, H_2CSi_2); 0.09 (dd, 1H, $J = 14.3$, 2.5 Hz, H_2CSi_2); 0.11 (s, 3H, MeSi); 0.14 (s, 3H, MeSi); 0.30 (d, 3H, $J = 3.7$ Hz, MeSiH); 0.41 (s, 3H, MeSi); 0.47 (s, 3H, MeSi); 2.16 (s, 3H, *p*-Me); 2.34 (s, 6H, *o*-Me); 4.54 (m, 1H, HSi); 6.70 (br s, 2H, mesityl ring protons); 6.80–7.02 (m, 5H, Ph). ^{13}C NMR ($CDCl_3$, 22.5 MHz): δ −5.7, −1.0, −0.5, 0.6, 4.0, 4.8, 20.9, 24.5, 125.1, 125.4, 127.3, 128.7, 133.4, 138.0, 143.8, 145.8, 163.3, 181.9. Anal. Found: C, 69.93; H, 8.67. $C_{23}H_{34}Si_3$ calcd.: C, 69.98; H, 8.68%.

Reaction of VII with hydrogen chloride followed by hydrolysis

A solution of 61.3 mg (0.156 mmol) of VII in 10 mL of benzene was placed in a 25 mL two necked flask. Dry hydrogen chloride gas was bubbled into the solution for 30 min with a rate of 1–2 mL/s. The resulting mixture was hydrolyzed with water and the organic layer was separated. The aqueous layer was extracted with ether. The organic layer and the extracts were combined and dried over potassium carbonate. The solvent was evaporated and the the residue was treated with preparative TLC (silica gel) to give pure VIII (23.2 mg, 51% yield): MS *m/e* 292 (M^+). IR (cm^{-1}): ν(O–H) 3264, ν(Si–H) 2125, ν(Si–O) 1070. ^1H NMR ($CDCl_3$, 90 MHz): δ −0.25 (dd, 1H, $J = 14.5$, 2.2 Hz, H_2CSi_2); −0.01 (s, 3H, MeSi); 0.05 (s, 3H, MeSi); 0.10 (dd, 1H, $J = 14.5$, 2.2 Hz, H_2CSi_2); 0.11 (s, 3H, MeSi); 0.16 (s, 3H, MeSi); 0.36 (d, 3H, $J = 3.5$ Hz, MeSiH); 4.65 (m, 1H, HSi); 6.90–7.35 (m, 5H, Ph). Exact MS calculated for $C_{14}H_{24}OSi_3$: 292.1134 (found: 292.1149). Anal. Found: C, 57.46; H, 8.24. $C_{14}H_{24}OSi_3$ calcd.: C, 57.47; H, 8.27%.

Reaction of VII with hydrogen chloride followed by methylation

A solution of 92.8 mg (0.236 mmol) of VII in 10 mL of benzene was placed in a 25 mL two necked flask. Dry hydrogen chloride gas was bubbled into the solution for 30 min with a rate of 1–2 mL/s. After evaporation of hydrogen chloride and a half volume of benzene under reduced pressure, 0.2 mL (0.3 mmol) of 1.5 *M* ether solution of methyl magnesium iodide was added. After hydrolysis with water, the organic layer was separated. The aqueous layer was extracted with hexane. The organic layer and the extracts were combined and dried over potassium carbonate. The solvent was evaporated and then the residue was treated with MPLC (silica gel, eluent hexane) to give 62.5 mg (91% yield) of IX. Analytically pure sample was obtained by preparative GLC: MS *m/e* 290 (M^+). IR ν(Si–H) 2107 cm^{-1}. ^1H NMR ($CDCl_3$, 90 MHz): δ −0.41 (dd, 1H, $J = 16.0$, 2.0 Hz, silacyclopentene ring proton); −0.27 (s, 9H, Me_3Si); −0.05 (s, 3H, MeSi); 0.02 (s, 3H, MeSi); 0.04 (dd, 1H, $J = 16.0$, 3.7 Hz, silacyclopentene ring proton); 0.21 (d, 3H, MeSi(H)); 4.48–4.59 (m, 1H, HSi); 6.72–7.17 (m, 5H, Ph). ^{13}C NMR ($CDCl_3$, 22.5 MHz): δ −5.9, −0.8, −0.7, −0.5, −0.2, 0.9, 125.5, 126.0, 127.7, 128.4, 146.7, 162.4. Exact MS calculated for $C_{15}H_{26}Si_3$: 290.1342 (Found: 290.1347).

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