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Thermal Generation of 2-Silanaphthalene Intermediates

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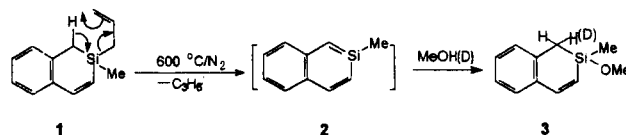
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There have been many reports of the generation and reaction of silabenzene intermediate, an attractive molecule in silicon chemistry, from the readily available chloro or allyl precursors.¹ The first generation of silatoluene intermediate formed *via* a thermally induced retro-ene extrusion of propene from 1-methyl-1-allyl-1-silacyclohexa-2,4-diene was reported by the Barton group,² and the existence was confirmed by matrix isolation method at low temperature.³ Thermal fragmentation of 1-methyl-2,3,4,5-tetraphenylsilacyclopentadienyl-diazomethane to give silatoluene was reported by Ando and Sekiguchi.⁴ West and Rich reported evidence for the existence of hexamethyl-1,4-disilabenzene intermediate produced by photochemical or thermal extrusions of anthracene from the precursor.⁵ The attempted generation of 9-silaanthracene from the suitable precursors has been reported.^{6,7a} However, in solution, the necessary elimination for formation of 9-silaanthracene from the chloro precursors fails to occur and reaction proceeds *via* intermolecular substitution leading to dimeric or polymeric products.^{6a,b} No evidence for the generation of 9-silaanthracene intermediate from photolysis of 10-diazo-9,9-dimethyl-9,10-dihydro-9-silaanthracene in methanol^{7a} and flash vacuum thermolysis

of 9,10-dibenzyl-9,10-diphenyl-9,10-dihydro-9-silaanthracene^{6c} was observed. There is a precedent for the unsuccessful approach to generate 2-silanaphthalene intermediate from the thermolysis or photolysis of diazo-2,2-diphenyl-4-bromo-1,2-dihydro-2-silanaphthalene (**4**) in the presence of methanol.⁷

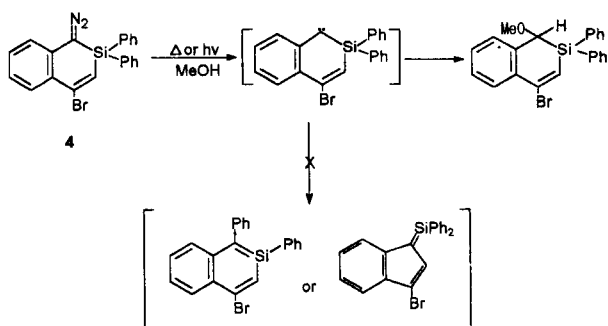
Recently, we reported the results of a thermolytic reaction of 2-allyl-2-methyl-1,2-dihydro-2-silanaphthalene (**1**) with methanol or methanol-*d*₁ in which appeared to involve generation of, an aromatic silicon-containing analogue of naphthalene, 2-methyl-2-silanaphthalene intermediate (**2**).⁸



We now wish to report the generation and trapping of 2-allyl- (**9**) and 2-methoxy-2-silanaphthalene (**11**) intermediates which could arise from the thermolytic reaction of 2,2-diallyl-1,2-dihydro-2-silanaphthalene (**7**) with methanol or methanol-*d*₁.

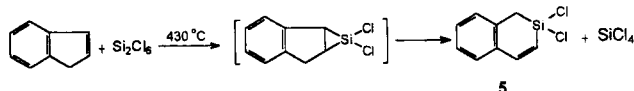
Results and Discussion

Attempts to generate 2-silanaphthalene by the thermolysis or photolysis of silyl diazo compound **4** have proved unsuccessful.⁷

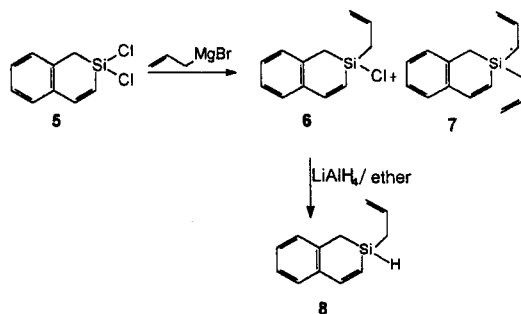


Recently, however, evidence for the existence of 2-silanaphthalene which could be generated from the coprolysis of **1** and methanol has been described.⁸ After we had performed these experiment, subsequent studies on the generation of other examples of 2-silanaphthalene derivative were carried out under thermal condition.

The precursor, 2,2-dichloro-1,2-dihydro-2-silanaphthalene (**5**),⁹ was produced in 35% yield from the coprolysis of hexachlorodisilane and indene at 430 °C with a continuous 55 mL/min flow of nitrogen gas.^{1a,8}

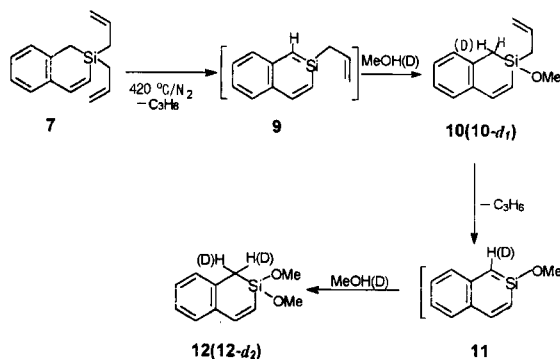


Compound **7** and **8** were prepared with the purpose of converting it by flow pyrolysis into 2-silanaphthalene intermediate. Reaction of **5** with an allylmagnesium bromide in dry ether produced 2-allyl-2-chloro- (**6**) and 2,2-diallyl-1,2-dihydro-2-silanaphthalene (**7**) in 15% and 50% yield, respectively. The product **8** was prepared with a yield of 89% by reducing 2-allyl-2-chloro-1,2-dihydro-2-silanaphthalene (**6**) with lithium aluminum hydride in dry ether.

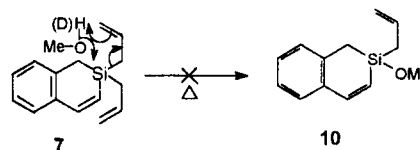


Pyrolysis of 2,2-diallyl-1,2-dihydro-2-silanaphthalene (**7**) in the presence of a twenty-fold excess of methanol which is a commonly used trapping agent for silicon-carbon doubly bonded intermediate^{5,11} was performed at 420 °C in a vertical quartz tube packed with quartz chips under the flow of nitrogen gas (55 mL/min). The two major products, 2-allyl-2-methoxy- (**10**) and 2,2-dimethoxy-1,2-dihydro-2-silanaphthalene (**12**), were produced in 39% and 20% yield, respectively *via* a retro-ene elimination of propene from **7**

and **10**. The observed products **10** (**10-d₁**) and **12** (**12-d₂**) can arise from the addition of methanol or methanol-*d*₁ into the 2-allyl- (**9**) and 2-methoxy-2-silanaphthalene (**11**) intermediates, respectively.

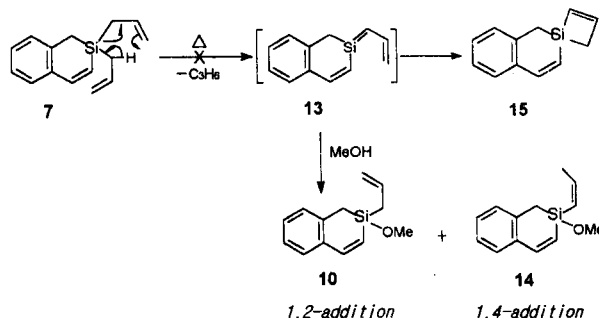


From these results it was thought that 2-silanaphthalenes were involved in the pyrolysis of **7** and methanol or methanol-*d*. However, an alternate pathway namely, the direct methanolysis, is possible. Note that in the latter mechanism shown below, the product **10** may be formed without involving 2-allyl-2-silanaphthalene (**9**).



A labelling experiment employing a methanol-*d*₁ may help identification of the reaction pathway for generation of 2-silanaphthalene intermediate. The coprolysis of **7** and MeOD at 420 °C (under N₂ flow) afforded a trapped adduct **10-d₁** arising from the replacement of hydrogen atom attached to the methylene carbon atom by a deuterium atom. This observation may rule out the latter reaction pathway involving the direct methanolysis and support the existence of intermediacy of 2-allyl- (**9**) and 2-methoxy-2-silanaphthalene (**11**).

The products **10**, **14**, and **15** can be derived from the addition (1,2 and 1,4) of methanol and electrocyclic reaction¹² of silabutadiene **13**. However, the formation of these products was not observed. The following reaction pathway is inefficient since this intermediate **13** loses its aromaticity.



The molecular ions (*M*⁺) of the target compounds (**6**, **7**, **8**, **10** and **12**) were clearly visible in their 70 eV electron ionization (EI) mass spectra. Unimolecular fragmentations of the molecular ion were initiated by the facile cleavage of bonds containing the silicon atom. The common frag-

Table 1. Partial EI Mass Spectra of 1,2-Dihydro-2-Silaphthalene Derivatives

Compound	6	7	8	10	12
X	allyl	allyl	allyl	allyl	OMe
Y	Cl	allyl	H	OMe	OMe
M ⁺ ^a	220(9) ^b	226(9)	186(13)	216(6)	206(68)
[M-X] ⁺	179(100)	185(36)	145(100)	175(100)	175(24)
[M-HX] ⁺ ^c	178(33)	184(19)	144(46)	174(6)	174(85)
[M-Y] ⁺	185(0)	185 ^d	185(0)	185(0)	175 ^d
[M-HY] ⁺	184(0)	184 ^c	184(0)	184(0)	174 ^c

^a Molecular ion. ^b m/z (% relative intensity). ^c The ionized 2-silaphthalene. ^d Same as [M-X]⁺. ^e Same as [M-HX]⁺.

mentation of the target compounds are suggested to be the loss of a substituent on the silicon atom from the molecular ions and the formation of ionized 2-silaphthalenes through the β -elimination of a substituent on the silicon atom and an adjacent hydrogen atom (Table 1).

From the underlined entries in Table 1 it is seen that ionized 2-silaphthalene intermediates occur in the mass spectra of all compounds (**6**, **7**, **8**, **10** and **12**). In parallel to the observation that the pyrolysis of 2,2-diallyl-1,2-dihydro-2-silaphthalene (**7**) produced the 2-silaphthalene intermediates (**9** and **11**), the ionized 2-silaphthalenes were observed in the mass spectra of the variously substituted 1,2-dihydro-2-silaphthalene. It appears that an allyl group among substituents of the target compounds was the most favorable one in the β elimination.

Experimental Section

General. For the synthesis of precursors, indene, hexachlorodisilane, allyl bromide, magnesium, and lithium aluminum hydride (LAH) were purchased from Aldrich Chemical Company and used without further purification. All pyrolyses were carried out in a vertical quartz tube (10 mm i.d. $\times$ 30 cm) packed with quartz chips under flow of nitrogen gas (55 mL/min). The progress of reaction was followed by gas chromatography with a flame ionization detector (FID) using a Hewlett-Packard 5890 instrument on a HP-1 capillary column (cross-linked 5% methyl phenyl silicone, 25 m). Separation of the products was performed on a Varian Model 920 GC with a thermal conductivity detector (TCD) using 20% OV-17 column (Chromosorb W 80/100 1/4 in $\times$ 3 ft). The ¹H NMR and ¹³C NMR spectra were recorded on a Hitachi R1200 60 MHz and Bruker AM-300 NMR spectrophotometer in CDCl₃ with tetramethylsilane (TMS) as an internal standard. Low resolution mass spectra were obtained by using a Hewlett Packard 5890 Series II GC coupled to a 5970 Series mass selective detector (electron energy: 70 eV). High resolution mass spectra were obtained by using a Jeol SX-102A double focusing mass spectrometer.

Preparation of 2,2-Dichloro-1,2-dihydro-2-silaphthalene (5). A solution of 4.6 g (39 mmol) of indene and 3.1 g (12 mmol) of hexachlorodisilane was injected slowly using a gas tight syringe (or dropping funnel) into a vertical quartz tube packed with quartz chips at 430 °C and operated with a flow of dry nitrogen gas regulated at a rate of 55 mL/min. The pyrolysate was swept through the reaction chamber with nitrogen gas into a trap cooled with dry

ice-acetone. The tar from the reaction mixture was filtered by using a celite-packed column, and unreacted indene was separated by vacuum distillation. The pure product was isolated by preparative GC (20% OV-17, 3ft) and afforded 0.9 g (4.2 mmol, 35%).

Compound (**5**); ¹H NMR (CDCl₃, 300 MHz): δ 2.50 (s, 2H, CH₂SiC=), 5.92 (d, 1H, SiCH=C, J =14.1 Hz), 6.88-6.98 (m, 4H, C₆H₄), 7.15 (d, 1H, SiC=CH, J =14.1 Hz). ¹³C NMR (CDCl₃): δ 24.63, 121.77, 127.14, 129.07, 131.98, 132.53, 133.10, 133.64, 152.41. MS m/z (rel. intensity): 218 (M⁺+4, 4), 216 (M⁺+2, 21), 214 (M⁺, 33), 180 (8), 179 (4), 178 (21), 143 (4), 116 (100), 115 (87), 100 (5), 98 (7), 89 (22), 65 (14), 63 (41).

Preparation of 2-Allyl-2-chloro-1,2-dihydro-2-silaphthalene (6) and 2,2-Diallyl-1,2-dihydro-2-silaphthalene (7). For the preparation of allylmagnesium bromide, a mixture of 0.6 g (25 mg atoms) of magnesium turnings and 30 mL of dry ether was placed in a 100 mL flask equipped with a mechanical stirrer, a reflux condenser, and a nitrogen inlet tube. A small piece of iodine was added in the cooled flask and a solution of 1.2 g (10 mmol) of allyl bromide in 20 mL of dry ether was added to the flask with vigorous stirring.¹⁰ The formed Grignard reagent was added dropwise to the flask which was charged with 0.5 g (2.3 mmol) of compound **5** and 30 mL of dry cyclohexane. The progress and formation of the products **6** and **7** were checked by analytical GC. Preparative GC afforded 76 mg (0.34 mmol, 15%) of product **6** and 0.26 g (1.15 mmol, 50%) of product **7**.

Compound (**6**); MS m/z (rel. intensity): 222 (M⁺+2, 3), 220 (M⁺, 9), 181 (37), 179 (100), 178 (33), 165 (2), 143 (10), 116 (4), 115 (22), 91 (2), 89 (6), 77 (1), 65 (14), 63 (36).

Compound (**7**); ¹H NMR (CDCl₃, 300 MHz): δ 1.70 (d, 4H, CH₂CH=CH₂, J =8.1 Hz), 2.18 (s, 2H, CH₂SiC=), 4.91 (m, 4H, CH₂CH=CH₂), 5.77 (m, 2H, CH₂CH=CH₂), 6.03 (d, 1H, SiCH=C, J =14.2 Hz), 7.06-7.12 (m, 4H, C₆H₄), 7.31 (d, 1H, SiC=CH, J =14.2 Hz). ¹³C NMR (CDCl₃, 300 MHz): δ 15.71, 20.29, 114.19, 123.98, 125.78, 127.63, 130.66, 132.14, 133.69, 135.24, 135.82, 148.81. MS m/z (rel. intensity): 226 (M⁺, 9), 185 (36), 184 (19), 157 (5), 145 (8), 144 (16), 143 (100), 131 (7), 119 (5), 117 (8), 116 (3), 115 (8), 105 (3), 83 (5), 69 (8), 53 (3). HRMS calcd for C₁₅H₁₈Si 226.1178, found 226.1173.

Preparation of 2-Allyl-1,2-dihydro-2-silaphthalene (8). A mixture of 0.3 g (1.36 mmol) of compound **6** and 0.19 g (5 mmol) of LiAlH₄ were reacted at room temperature in dry ether. After LiAlH₄ was filtered through celite-packed column, solvent was evaporated. Product **8** was separated by preparative GC and afforded 0.23 g (1.24 mmol, 89%).

Compound (**8**); ¹H NMR (CDCl₃, 300 MHz): 1.72-1.77 (m, 2H, CH₂CH=CH₂), 2.26-2.31 (m, 2H, CH₂SiC=), 4.19-4.30 (m, 1H, SiH), 4.89-5.00 (m, 2H, CH₂CH=CH₂), 5.71-5.93 (m, 1H, CH₂CH=CH₂), 6.07 (d, 1H, SiCH=C, J =14.2 Hz), 7.13 (bs, 4H, C₆H₄), 7.34 (d, 1H, SiC=CH, J =14.2 Hz). ¹³C NMR (CDCl₃, 300 MHz): δ 14.25, 19.50, 114.28, 121.67, 125.92, 127.76, 130.72, 132.03, 133.56, 134.28, 134.59, 149.13. MS m/z (rel. intensity): 186 (M⁺, 13), 158 (3), 145 (100), 144 (46), 143 (52), 142 (5), 131 (3), 119 (19), 118 (3), 117 (13), 116 (4), 115 (13), 105 (10), 93 (7), 91 (4), 89

(3), 77 (3), 67 (6), 53 (14). HRMS calcd for $C_{12}H_{14}Si$ 186.0865, found 186.0871.

Pyrolysis of 7 and Methanol. A mixture of 0.3 g (1.33 mmol) of 7 and 0.84 g (26 mmol) of methanol as a trapping agent was injected slowly into the vertical quartz tube packed with quartz chips using a gas tight syringe under flow nitrogen gas (55 mL/min). The pyrolysate was collected in two traps cooled with dry ice-acetone and liquid nitrogen. Bulb to bulb distillation of the pyrolysate followed by preparative GC on an OV-17 column afforded 0.11 g (0.51 mmol, 39%) of product 10 and 56 mg (0.27 mmol, 20%) of product 12. The spectral properties of the two products are given below.

Compound (10); 1H NMR ($CDCl_3$, 300 MHz): δ 1.81 (d, 2H, $CH_2CH=CH_2$, $J=8.1$ Hz), 2.21 (center of AB quartet, 2H, $CH_2SiC=$, $J=16.8$ Hz $\nu_A=2.28$, $\nu_B=2.13$), 3.42 (s, 3H, SiOMe), 4.91-5.01 (m, 2H, $CH_2CH=CH_2$), 5.76-5.88 (m, 1H, $CH_2CH=CH_2$), 6.07 (d, 1H, $SiCH=C$, $J=14.4$ Hz), 7.08-7.17 (m, 4H, C_6H_4), 7.45 (d, 1H, $SiC=CH$, $J=14.4$ Hz). ^{13}C NMR ($CDCl_3$, 300 MHz): δ 17.45, 21.46, 50.46, 114.65, 122.87, 125.76, 127.75, 130.94, 132.09, 132.81, 134.90, 135.59, 151.14. MS m/z (rel. intensity): 216 (M^+ , 6), 175 (100), 174 (6), 145 (10), 144 (2), 143 (8), 131 (2), 119 (3), 117 (5), 116 (3), 115 (9), 105 (2), 89 (2), 77 (1), 65 (1), 63 (2), 59 (96) 53 (2). HRMS calcd for $C_{13}H_{16}SiO$ 216.0970, found 216.0964.

Compound (12); 1H NMR ($CDCl_3$, 300 MHz): δ 2.22 (s, 2H, $CH_2SiC=$), 3.50 (s, 6H, (SiOMe) $_2$), 6.02 (d, 1H, $SiCH=C$, $J=14.7$ Hz), 7.05-7.11 (m, 4H, C_6H_4), 7.45 (d, 1H, $SiC=CH$, $J=14.7$ Hz). ^{13}C NMR ($CDCl_3$, 300 MHz): δ 16.53, 50.28, 121.66, 125.88, 127.68, 131.10, 132.43, 134.95, 135.37, 153.00. MS m/z (rel. intensity): 206 (M^+ , 68), 176 (15), 175 (24), 174 (85), 161 (4), 146 (4), 145 (6), 144 (12), 143 (8), 131 (3), 130 (8), 117 (11), 116 (100), 115 (52), 91 (25), 89 (9), 65 (3), 63 (4), 59 (55). HRMS calcd for $C_{11}H_{14}SiO_2$ 206.0763, found 206.0760.

Pyrolysis of 7 and Methanol- d_1 . A mixture of 0.3 g (1.33 mmol) of 7 and 0.86 g (26 mmol) of methanol- d_1 as a trapping agent was injected slowly into the vertical quartz tube packed with quartz chips using a gas tight syringe under flow nitrogen gas (55 mL/min). The pyrolysate was collected in two traps cooled with dry ice-acetone and liquid nitrogen. Bulb to bulb distillation of the mixture followed by preparative GC on the same OV-17 column afforded the two trapping adducts. The two major products, 10- d_1 (0.49 mmol, 37%) and 12- d_2 (0.25 mmol, 19%), were identified on the basis of their spectroscopic data.

Compound (10- d_1); 1H NMR ($CDCl_3$, 300 MHz): δ 1.81 (d, 2H, $CH_2CH=CH_2$, $J=8.1$ Hz), 2.21 (bs, 1H, $CHDSiC=$), 3.42 (s, 3H, SiOMe), 4.91-5.01 (m, 2H, $CH_2CH=CH_2$), 5.76-5.88 (m, 1H, $CH_2CH=CH_2$), 6.07 (d, 1H, $SiCH=C$, $J=14.4$ Hz), 7.08-7.17 (m, 4H, C_6H_4), 7.45 (d, 1H, $SiC=CH$, $J=14.4$ Hz). ^{13}C NMR ($CDCl_3$, 300 MHz): δ 17.45, 21.46, 50.46, 114.65, 122.87, 125.75, 130.94, 132.09, 132.81, 134.90, 135.59, 151.14. MS m/z (rel. intensity): 217 (M^+ , 6), 187 (7), 176 (77), 175 (34), 174 (4), 146 (14), 145 (30), 144 (11), 143 (14), 131 (13), 119 (8), 118 (3), 117 (12), 116 (15), 115 (18), 105 (13), 91 (9), 89 (6), 77 (7), 69 (6), 63 (6), 59 (100), 53 (8). HRMS calcd for $C_{13}H_{15}DSiO$

217.1032, found 217.1020.

Compound (12- d_2); 1H NMR ($CDCl_3$, 300 MHz): δ 3.50 (s, 6H, (SiOMe) $_2$), 6.02 (d, 1H, $SiCH=C$, $J=14.7$ Hz), 7.05-7.11 (m, 4H, C_6H_4), 7.45 (d, 1H, $SiC=CH$, $J=14.7$ Hz). ^{13}C NMR ($CDCl_3$, 300 MHz): δ 16.53, 50.28, 121.66, 125.88, 127.68, 131.10, 132.43, 134.95, 135.37, 153.00. MS m/z (rel. intensity): 208 (M^+ , 21), 178 (4), 177 (8), 175 (23), 163 (2), 161 (4), 148 (2), 147 (4), 146 (6), 145 (16), 144 (6), 143 (9), 132 (4), 119 (17), 118 (47), 117 (48), 116 (33), 115 (20), 105 (11), 91 (19), 77 (6), 63 (7), 59 (100). HRMS calcd for $C_{11}H_{12}D_2SiO_2$ 208.0886, found 208.0875.

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