

Photoinduced Rearrangement Reaction of 2-(Pentamethyldisilanyloxymethyl)phenylpentamethyldisilane[†]

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It is well known that disilane molecules having a silicon-silicon σ bond show unique properties in the photochemical¹⁻⁴ and thermal reactions⁵ and in the transition metal catalyzed reactions⁶⁻¹⁴ since the first reports on the unusual electronic spectra of arylsilanes by Sakurai *et al.*,¹⁵ Gilman *et al.*¹⁶ and Hague *et al.*¹⁷ in 1964.

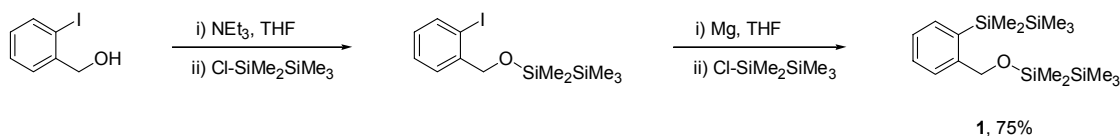
In the context of our interest on the synthesis of silicon containing heterocyclic compounds by the photoreactions of *ortho*-substituted arylalkynylsilanes¹⁸⁻²⁹ and arylsilanes,^{30,31} we have very recently reported on the photoreaction of (2-pentamethyldisilanyloxy)phenylpentamethyldisilane³² in the presence of methanol or acetone affording a novel intramolecular photoproduct *via* silene intermediate. In light of a reactivity of benzyl position, we have now extended our research on the compound 2-(pentamethyldisilanyloxymethyl)phenylpentamethyldisilane **1** since novel photoproducts from the intramolecular reaction between two *ortho*-pentamethyldisilanyl groups are expected. In this article, we would like to report the detailed photochemical study of 2-(pentamethyldisilanyloxymethyl)phenylpentamethyldisilane **1**.

The starting 2-(pentamethyldisilanyloxymethyl)phenylpentamethyldisilane **1** was prepared by the reaction of chloro-pentamethyldisilane with 2-(pentamethyldisilanyloxymethyl)-

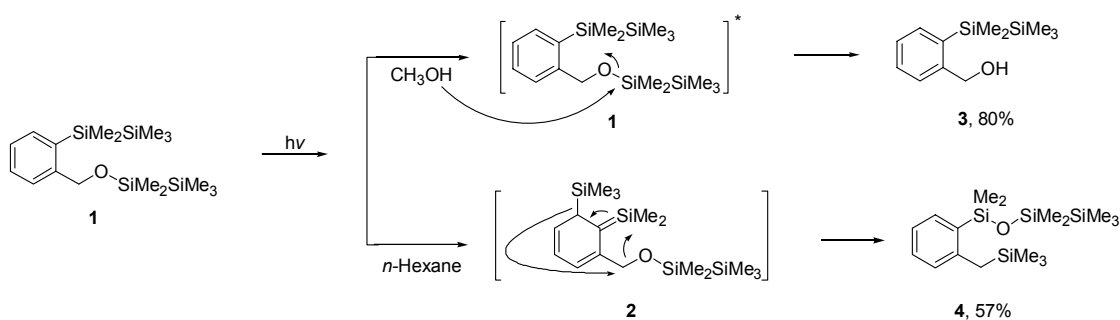
phenylmagnesium iodide in tetrahydrofuran (Scheme 1).

To investigate whether or not the pentamethyldisilanyloxymethyl group as *ortho* substituent in **1** reacts intramolecularly with the photoexcited state of **1**, the photolysis of **1** was performed.

Irradiation of **1** in deaerated methanol with 254 nm UV light afforded a compound **3** (80% yield) along with some decomposition products of unknown structure as shown in Scheme 2, when 98% of **1** was photolyzed. The formation of a photoproduct **3** can best be explained in terms of the nucleophilic attack at α silicon atom of pentamethyldisilanyloxymethyl group in the photoexcited state of **1** by methanol (path d). Indeed, it is well known that the photolysis of substituted phenylpentamethyldisilanes has four reaction pathways:³³ (path a) formation of a silene compound and a trimethylsilane,³⁴ (path b) formation of a silatriene compound arising from 1,3-shift of trimethylsilyl radical, which is formed *via* homolytic cleavage of silicon-silicon bond to the C₆ position of benzene ring,³⁵⁻⁴⁶ (path c) formation of a trimethylsilyl compound *via* elimination of a silylene,⁴⁷ (path d) formation of a direct solvolysis compound accompanied by the formation of trimethylalkoxysilane and trimethylsilane.^{48,49} In relation to the photoreactions of the compounds with bis(disilanyl) group similar to the compound

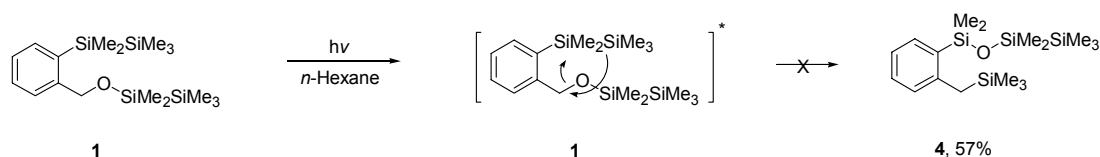


Scheme 1



Scheme 2

[†]This paper is dedicated to Professor Sunggak Kim on the occasion of his honorable retirement.



Scheme 3

1, Ishikawa *et al.*⁵⁰ found the photolysis of 1,2-, 1,3-, and 1,4-bis(disilanyl)benzene in the presence of isobutene gave 2-(isobutyldimethylsilyl)-1-(pentamethyldisilanyl)-3-(trimethylsilyl)benzene, 1,1-dimethyl-6-[1,1-dimethyl-2-(trimethylsilyl)ethyl]-4-(pentamethyldisilanyl)silolepin, and 4-(isobutyldimethylsilyl)-1-(pentamethyldisilanyl)-5-(trimethylsilyl)benzene *via* the photochemically generated rearranged silatriene intermediate (path b). In the case of the photolysis of 1,4-bis(pentamethyldisilanyl)naphthalene⁵¹ in the presence of methanol, 4-(dimethylsilyl)-1-(pentamethyldisilanyl)-5-(trimethylsilyl)naphthalene *via* the radical scission of a silicon-silicon bond of the photoexcited starting compound and 6-(methoxydimethylsilyl)-6,7-bis(trimethylsilyl)-5,8-(dimethylsilano)-5,6,7,8-tetrahydronaphthalene *via* a silatriene intermediate (path b) were obtained. And, in the case of the photoreaction of 2-(pentamethyldisilanyloxy)phenylpentamethyldisilane³² in the presence of methanol, the photoproducts *via* silatriene intermediate (path b) and direct solvolysis (path d) were obtained.

Irradiation of **1** in deaerated *n*-hexane with 254 nm UV light afforded a novel rearranged photoproduct **4** (57% yield) along with some decomposition products of unknown structure as shown in Scheme 2, when 85% of **1** was photolyzed. The formation of a novel photoproduct **4** can best be explained in terms of the initial formation of silatriene intermediate **2** (path b) arising from 1,3-migration of trimethylsilyl radical, which is formed *via* the homolytic cleavage of silicon-silicon bond of pentamethyldisilanyl group in the photoexcited state of **1** to the C₆ position of benzene ring. The intramolecular oxygen attack of pentamethyldisilanyloxymethyl group in **2** to silicon atom in silene moiety of intermediate **2** accompanied by an aromatization, 1,4-trimethylsilyl shift and the elimination of pentamethyldisilanyloxy group gives the compound **4**. In relation to the formation of the photoproduct **4**, our laboratory reported the photolysis of (2-acetoxymethylphenyl)pentamethyldisilane⁵² in the presence of methanol gave the rearranged photoproduct *via* the intermolecular reaction of methanol to silicon atom in silatriene moiety of reaction intermediate (path b) accompanied by an aromatization, 1,4-trimethylsilyl shift similar to the formation of the compound **4**. One might consider the possibility that the photoproduct **4** could be produced by a concerted process involving the intramolecular oxygen attack of the pentamethyldisilanyloxy group to α silicon atom of the pentamethyldisilanyl group in the photoexcited state of **1** accompanied by the attack of trimethylsilyl group to a benzyl position of the photoexcited state of **1** as shown in Scheme 3. However, no product derived from the intramolecular reaction of other possible intermediates except silatriene intermediate with *ortho*-substituted group was detected in the photolysis of aryl-disilanes^{30-32,52} and the reaction mechanism as shown in Scheme 3 are not reasonable. The structure of the photoproduct **4** was

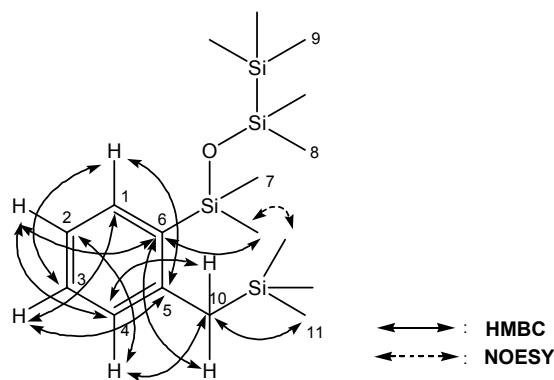


Figure 1. Important correlations observed in HMBC and NOESY spectra of photoproduct **4**.

also determined by various physical methods, such as ¹H NMR, ¹³C NMR, 2D NMR (¹H-¹H, ¹H-¹³C COSY, HMBC, and NOESY), UV, FT-IR, and high resolution mass spectrometry. The ¹H-¹H and ¹H-¹³C correlation spectroscopy (COSY) spectra of **4** were carried out to identify the protons directly attached to the individual carbons. According to the correlated peaks in **4**, we have been able to identify the pairs of carbons and directly bonded protons as shown in Table 1. From the HMBC spectrum of **4**, the structure skeleton of **4** was determined. The connectivity of the carbon C(6) to the dimethylsilyl group in **4** was established by the presence of the cross-peaks due to the vicinal coupling between the quaternary carbon C(6) and the protons of C(7) in **4**. The presence of the cross-peaks due to the vicinal coupling between the carbon C(10) and the proton of C(4), and the carbon C(10) and the protons of C(11) in **4** showed the connectivity of the carbon C(5) to the trimethylsilylmethyl group in **4**. In the same manner, the presence of the cross-peaks due to the vicinal coupling between the quaternary carbon C(6) and the protons of C(10) in **4** also showed the connectivity of the carbon C(5) to the trimethylsilylmethyl group in **4**. From the presence of the cross-peaks between the protons of C(7) and the protons of C(11) which is showed in a nuclear overhauser and exchange spectroscopy (NOESY) of **4**, it was determined that these protons are located on the same side of the molecule. Thus, the skeletal structure of **4** was unambiguously established as shown in Figure 1.

In conclusion, the photolysis of **1** in methanol provided a compound **3**. The formation of **3** can be explained in terms of α silicon atom attack of methanol to pentamethyldisilanyloxymethyl group in the photoexcited state of **1**. Irradiation of **1** in *n*-hexane gave a novel rearranged photoproduct **4**. The formation of **4** in *n*-hexane solvent was probably explained by the intramolecular oxygen attack to silicon atom in silatriene

Table 1. ^1H NMR (500 MHz), ^{13}C NMR (125 MHz), and HMBC data for photoproduct **4** in CDCl_3^a

Position	δ_c (ppm)	M^b	δ_H (ppm)	I^c	M^d	J_{H-H} (Hz)	HMBC ^e
1	134.9	d	7.51	1H	dd	7.5 (J_{1H-2H}), 1.5 (J_{1H-3H})	H3
2	123.6	d	7.10	1H	d	7.5 (J_{2H-1H})	H4
3	129.4	d	7.26	1H	td	7.5 ($J_{3H-2H,4H}$), 1.5 (J_{3H-1H})	H1
4	128.5	d	7.06	1H	d	7.5 (J_{4H-3H})	H2, H10
5	146.4	s					H1, H3
6	137.3	s					H2, H4, H7, H10
7	3.41	q	0.39	3H	s		
8	2.29	q	0.24	3H	s		
9	-2.02	q	0.09	3H	s		
10	26.7	t	2.35	2H	s		H4, H11
11	-0.59	q	0.05	3H	s		H10

^aAll these assignments were confirmed by ^1H - ^1H and ^1H - ^{13}C COSY and NOESY, HMBC spectra. ^bMultiplicities were determined by DEPT spectrum. ^cIntegrated intensity. ^dMultiplicities. ^eProtons correlated to carbon resonances in ^{13}C column.

moiety, aromatization, 1,4-trimethylsilyl shift, and the elimination of pentamethyldisilyloxy group.

Experimental

General methods. All reactions were carried out under an atmosphere of dry nitrogen. ^1H and ^{13}C NMR spectra were recorded on Bruker AM-300 and Bruker AC-200 spectrometers with chemical shifts being referenced against TMS as an internal standard or the signal of the solvent CDCl_3 . UV absorption spectra were recorded on a Hewlett-Packard 8453 spectrophotometer. Mass spectra were determined at 70 eV with a Hewlett-Packard 5985A GC-MS by the electron impact (EI) method. FT-IR spectra were recorded on a Bomem MB-100 spectrometer in KBr pellets and NaCl cell. High-performance liquid chromatography was performed on a Waters Associates Model 244 liquid chromatograph (Mildford, MA) equipped with a Model 6000A solvent delivery system, Model 440 UV absorbance detector fixed at 254 nm, and Model U6K universal injector. Lichrosorb SI-60 column was used for preparative analyses. Thin layer chromatography (TLC) was performed on Sigma-Aldrich pre-coated silica gel F₂₅₄ aluminum foils. Purification of the reaction products was carried out by flash column chromatography using a glass column dry packed with silica gel (230 - 400 mesh ASTM).

Materials. Tetrahydrofuran was refluxed over sodium benzophenone and distilled under nitrogen. Methanol (HPLC grade) and *n*-hexane (HPLC grade) were distilled from CaH_2 before use. Et_3N was distilled from CaH_2 and stored over KOH pellets. Solvents of reagent grade for chromatography were used without further purification. Spectroscopic grade solvents were used for HPLC and UV absorption spectra.

Synthesis of 2-(pentamethyldisilyloxyethyl)phenylpentamethyldisilane 1. Triethylamine (0.71 mL, 5.12 mmol) was added to a solution of 2-iodobenzyl alcohol (1 g, 4.27 mmol) in tetrahydrofuran (30 mL) at room temperature under nitrogen atmosphere. Chloropentamethyldisilane (1 mL, 5.12 mmol) was added dropwisely to the resulting solution and the mixture was refluxed for 3 h. The reaction mixture was filtered with celite, and concentrated in vacuo. To this reaction residue, THF (30

mL) was added. This resulting reaction solution was added to Mg (0.31 g, 12.8 mmol) at room temperature under nitrogen atmosphere. Chloropentamethyldisilane (1 mL, 5.12 mmol) was added dropwisely to the resulting solution and the mixture was refluxed for 0.5 h. The reaction mixture was filtered with celite, and concentrated in vacuo. Water (30 mL) was added to the solution and extracted with ethyl acetate (3 × 15 mL). The combined ethyl acetate solution was washed with water (20 mL), brine (20 mL) and dried (MgSO_4). The solvent was evaporated in vacuo to give the crude product. Flash column chromatography with *n*-hexane/ethyl acetate (50/1, v/v) as an eluent gave **1** (1.18 g, 75% yield) as a colorless oil; ^1H NMR (CDCl_3 , 300 MHz) δ_H 0.082 (9H, s), 0.14 (9H, s), 0.29 (6H, s), 0.39 (6H, s), 4.71 (2H, s), 7.23 (1H, d, $J = 7.2$ Hz), 7.37 (1H, t, $J = 7.8$ Hz), 7.42 (1H, d, $J = 7.2$ Hz), 7.52 (1H, d, $J = 7.8$ Hz); ^{13}C NMR (CDCl_3 , 75 MHz) δ_C -2.09, -1.85, -1.59, -0.58, 65.8, 126.2, 126.4, 128.9, 134.5, 135.8, 146.7; UV (CH_2Cl_2) λ_{max} 236 nm; FT-IR (NaCl) 3057.6, 2951.2, 2894.1, 1436.4, 1246.7, 1091.5, 834.2, 800.6, 766.7 cm^{-1} ; MS (70 eV) m/z 368 (M^+); MS m/z (%) 368 (M^+ , 0.1), 295(10), 243(5), 208(100), 164(12), 132(5), 73(38); HRMS (M^+) calcd for $\text{C}_{17}\text{H}_{36}\text{OSi}_4$ 368.1843, found 368.1881.

Irradiation of 2-(pentamethyldisilyloxyethyl)phenylpentamethyldisilane 1 in methanol. A solution (5×10^{-4} M) of 2-(pentamethyldisilyloxyethyl)phenylpentamethyldisilane **1** (184 mg) in methanol (1 L) was deaerated by nitrogen purging for 1 h and irradiated in a Rayonet photochemical reactor, model RPR-208, equipped with RUL 254 nm lamps. After irradiation for 1 min, the resulting photoreaction mixture was concentrated in vacuo. The photoproduct **3** was isolated in (95.2 mg, 80% yield) in addition to 2% (3.7 mg) of the starting material **1** by column chromatography with *n*-hexane/ethyl acetate (20/1, v/v) as an eluent followed by normal phase HPLC using *n*-hexane/ethyl acetate (10/1, v/v) as an eluent. **3**; ^1H NMR (CDCl_3 , 300 MHz) δ_H 0.09 (9H, s), 0.43 (6H, s), 1.90 (1H, s), 4.71 (2H, s), 7.30 (1H, d, $J = 7.2$ Hz), 7.39 (1H, td, $J = 7.2$, 1.2 Hz), 7.49 (2H, m); ^{13}C NMR (CDCl_3 , 75 MHz) δ_C -1.87, -1.51, 65.9, 127.3, 127.8, 129.4, 135.1, 137.6, 146.4; UV (CH_2Cl_2) λ_{max} 236 nm; FT-IR (NaCl) 3325.2, 3100.7, 3024.7, 2914.2, 1558.0, 1215.8, 1110.8, 858.5, 816.5 cm^{-1} ; MS (70

eV) m/z 238 (M^+); MS m/z (%) 238 (M^+ , 6), 208(27), 179(28), 165(54), 147(100), 104(37), 83(23); HRMS (M^+) calcd for $C_{12}H_{22}OSi_2$ 238.1209, found 238.1234.

Irradiation of 2-(pentamethyldisilanyloxymethyl)phenylpentamethyldisilane 1 in *n*-hexane. A solution (5×10^{-4} M) of 2-(pentamethyldisilanyloxymethyl)phenylpentamethyldisilane **1** (184 mg) in *n*-hexane (1 L) was deaerated by nitrogen purging for 1 h and irradiated in a Rayonet photochemical reactor, model RPR-208, equipped with RUL 254 nm lamps. After irradiation for 40 min, the resulting photoreaction mixture was concentrated in vacuo. The photoproduct **4** was isolated in (104.9 mg, 57% yield) in addition to 15% (27.6 mg) of the starting material **1** by column chromatography with *n*-hexane/ethyl acetate (70/1, v/v) as an eluent followed by normal phase HPLC using *n*-hexane/ethyl acetate (40/1, v/v) as an eluent. **4**; UV (CH_2Cl_2) λ_{max} 283, 275 nm; FT-IR (NaCl) 3056.1, 2952.9, 2894.9, 1588.4, 1434.5, 1249.5, 1049.3, 833.7 cm^{-1} ; MS (70 eV) m/z 368 (M^+); MS m/z (%) 368 (M^+ , 4), 339(5), 295(6), 281(17), 265(12), 221(16), 207(100), 191(13), 147(25), 130(15), 73(47); HRMS (M^+) calcd for $C_{17}H_{36}OSi_4$ 368.1843, found 368.1892.

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