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Palladium-catalyzed hydrosilylation polymerization of dihydrosilanes with diynes affording silylene-divinylene polymers

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Abstract

The Pd-PCy$_3$ (Cy = cyclohexyl) system effectively catalyzed hydrosilylation polymerization of dihydrosilanes with *p*- or *m*-diethynylbenzene to give silylene-divinylene polymers with Ph, Me, and/or vinyl substituents at the silicon atoms. The resulting polymers showed characteristic absorption and fluorescence spectra in ultraviolet to visible region. *p*-Phenylene polymers displayed λ_{max} peaks in longer wavelength region than the corresponding *m*-phenylene polymers. In TGA, the 5% weight loss temperatures (Td$_5$) ranged from 436 to 501 °C, indicating rather high thermal stability of these polymers. Vinyl group-containing polymers exhibited higher Td$_5$ values and char yields than other polymers.

Keywords: Hydrosilylation; Polymerization; Palladium catalyst

1. Introduction

Organosilicon polymers have attracted increasing attention for their applicability to new functional materials [1,2]. Recently, some poly- or oligocarbosilanes with vinylene and/or arylene units in the backbones have been demonstrated to have intriguing physicochemical properties, which would be applicable to heat-resistant materials, ceramics, optoelectric devices, etc. [3–17]. Hydrosilylation polymerization using Pt [3–7] or Rh [8–16] catalysts is a versatile method for these poly- or oligocarbosilane synthesis. Some silylene-divinylene polymers were previously prepared in the Pt-catalyzed reactions of dihydrosilanes with diynes [5], although the molecular weights were generally as low as several thousands. In the course of our study on Pd-catalyzed hydrosilylation [17], we have found that Pd-PCy$_3$ catalyst is effective to prepare the silylene-divinylene polymers with relatively high molecular weights. In addition, different from the conventional Pt-catalyzed reaction, the Pd-catalyzed hydrosilylation is highly selective for C≡C bonds over C=C bonds, and therefore, modifiable vinyl group-substituted silicon polymers can be obtained by using a vinyldihydrosilane monomer. The resulting polymers exhibit unique photophysical and thermal properties depending on the backbone and/or substituent structures.

2. Results and discussion

Typically, when PhMeSiH$_2$ (**1a**) was allowed to react with *p*-diethynylbenzene (**2a**, 1 equiv.) in the presence of Pd$_2$(dba)$_3$-PCy$_3$ (dba = dibenzylideneacetone, 0.6 mol% Pd, P/Pd = 2) at 70 °C for 0.5 h, ^{1}H NMR measurement showed complete conversion of the starting monomers and formation of a 1:1 addition polymer **3a** (Scheme 1, Table 1). Purification by precipitation from benzene–2-propanol gave **3a** with satisfactory analytical and spectral data in 86% yield. The molecular weight M_w(M_w/M_n) was estimated by GPC at 49,000 (6.6) using polystyrene standards. The GPC profile showed a multimodal distribution of the molecular weight with shoulder-like peaks ranging from 10^3 to 10^6 (Fig. 1). In ^{29}Si NMR, three peaks were observed at − 15.7, − 15.6, and − 13.4 ppm (Fig. 2). A model reaction of **1a** with phenylacetylene (**4**, 2 equiv.) under similar conditions gave (β,β)- and (β,α)-type addition products,

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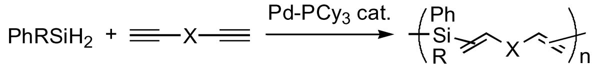


1a: R = Me
1b: R = Ph
1c: R = CH=CH2

2a: X = *p*-C6H4
2b: X = *m*-C6H4

3a: R = Me, X = *p*-C6H4
3b: R = Me, X = *m*-C6H4
3c: R = Ph, X = *p*-C6H4
3d: R = Ph, X = *m*-C6H4
3e: R = CH=CH2, X = *p*-C6H4
3f: R = CH=CH2, X = *m*-C6H4

Scheme 1.

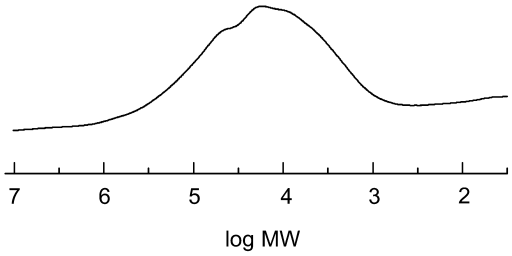


Fig. 1. GPC profile of **3a**; MW = molecular weight with reference to polystyrene standards.

PhMeSi(CH=CHPh)$_2$ (**5ββ**, ^{29}Si NMR: − 15.7 ppm) and PhMeSi(CH=CHPh)[C(=CH$_2$)Ph] (**5βα** − 13.5 ppm), in the ratio of 80:20 (totally ≥ 90% NMR yield, Scheme 2). The formation of the (α,α)-type isomer was negligible. Accordingly, the signals of − 15.7 and − 15.6 ppm for **3a** seemed to arise from the –CH=CH–Si–CH=CH– (β,β) linkages, while the signal of − 13.4 ppm would be attributed to the –CH=CH–Si–C(=CH$_2$)– (β,α) linkages. On the basis of the ^{1}H and ^{29}Si NMR integral curves, the ratio of (β,β):(β,α) was estimated at 78:22. Predominant formation of the (β,β) linkage was previously reported in a Pt-catalyzed reaction as well [5], in which the ratio of (β,β):(β,α):(α,α) was estimated at 60:26:14. As for the stereochemistry of the vinylene linkages of **3a**, a broad doublet-like signal with a rather large coupling constant (J = ca.19 Hz) was observed for the Si–CH=C vinylene proton in ^{1}H NMR. In addition, *trans* vinylene isomers for **5ββ** and **5βα** (J = 19.1 Hz for both isomers) were exclusively formed in the above model reaction. The results suggest that the vinylene moieties of **3a** have essentially *trans* geometry. On the other hand, although the terminal groups of **3a** have not been clearly determined, NMR and IR spectra suggested the presence of small amounts of Si–H groups (5.05–5.12 ppm in ^{1}H NMR, 2124 cm^{-1} for $\nu_{\text{Si-H}}$ in IR). In IR spectra, no broad peaks of Si–OH and Si–O–Si (around 1100–900 cm^{-1}) were observed, indicating that further conversion of the Si–H bonds by oxidation and/or hydrolysis were nearly negligible.

Likewise, Ph$_2$SiH$_2$ (**1b**) underwent the reaction with **2a** to give the corresponding polymer **3c** (M_w = 53,000, M_w/M_n = 6.8) in 90% yield, although the reactivity of **1b** was somewhat lower than that of **1a**. In addition to **2a**, its *meta* analog **2b** also reacted with **1a** or **1b** to give a 1:1 addition polymer **3b** (M_w = 20,000, M_w/M_n = 4.2, 85% yield) or **3d** (M_w = 12,000, M_w/M_n = 3.6, 94%), respectively. In these reactions, the *para* diyne **2a** showed higher reactivity and gave higher molecular weight polymers than the *meta* form **2b**. In place of the Pd$_2$(dba)$_3$-PCy$_3$ system, PdCl$_2$(PCy$_3$)$_2$ could be used as well, although higher temperature and/or longer reaction time were required; **1b** reacted with **2a** in the presence of PdCl$_2$(PCy$_3$)$_2$ at 90 °C for 18 h to give **3c** with M_w(M_w/M_n) being 12,000 (2.6) in 80% yield.

The Si–H bond addition by the Pd-PCy$_3$ catalyst is highly selective for C≡C bonds over C=C bonds. For instance, insertion of phenylacetylene into the Si–H bonds of PhSiH$_3$ smoothly proceeded in the presence of Pd$_2$(dba)$_3$-PCy$_3$ catalyst at room temperature [17], while styrene did not react at all even at 80 °C. Therefore, dihydrosilane monomers with vinyl groups are expected to undergo hydrosilylation polymerization with the vinyl groups being intact. In fact, the reaction of a vinyldihydrosilane, (CH$_2$=CH)PhSiH$_2$ (**1c**), with **2a** or **2b** provided the corresponding vinyl pendant group-containing *p*- or *m*-phenylene polymer **3e** (M_w = 20,000, M_w/M_n = 4.6, 91% yield) or **3f** (M_w = 12,000, M_w/M_n = 3.8, 88%), respectively.

The *p*-phenylene polymers **3a,c,e** showed UV/vis

Table 1
Pd-catalyzed reactions of dihydrosilanes **1** with diynes **2**

Run	1	2	Temp (°C)	Time (h)	3 (Yield,[a] %)	M_w(M_w/M_n)[b]	(β,β):(β,α)[c]
1	**1a**	**2a**	70	0.5	**3a** (86)	49,000 (6.6)	78:22
2	**1a**	**2b**	70	2	**3b** (85)	20,000 (4.2)	78:22
3	**1b**	**2a**	70	1	**3c** (90)	53,000 (6.8)	82:18
4	**1b**	**2b**	70	8	**3d** (94)	12,000 (3.6)	79:21
5[d]	**1c**	**2a**	110	6	**3e** (91)	20,000 (4.6)	75:25
6[d]	**1c**	**2b**	110	9	**3f** (88)	12,000 (3.8)	70:30

Reaction conditions: **1** 0.40 mmol, **2** 0.40 mmol, Pd$_2$(dba)$_3$-PCy$_3$ (0.0024 mmol Pd, P/Pd = 2), benzene-d_6 or benzene 0.40 ml.

[a] Yield after purification by precipitation from benzene–2-propanol.

[b] Estimated by GPC using polystyrene standards.

[c] Estimated by ^{1}H and/or ^{29}Si NMR.

[d] PdCl$_2$(PCy$_3$)$_2$ was used in place of Pd$_2$(dba)$_3$-PCy$_3$.

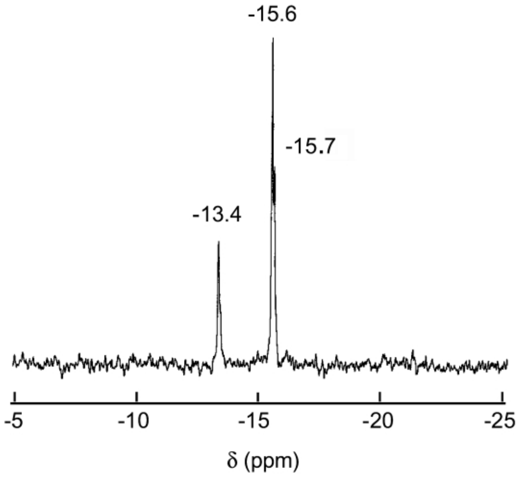
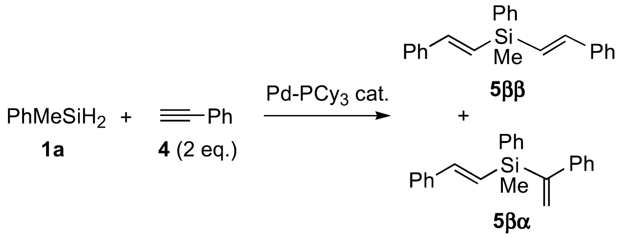


Fig. 2. ^{29}Si NMR spectra of **3a** (in benzene-d_6).



Scheme 2.

absorption peaks at around 305 nm in chloroform, while the corresponding *m*-phenylene polymers **3b,d,f** with shorter π-conjugation systems displayed blue-shifted peaks at around 260 nm (Table 2). Typical absorption spectra are shown as solid lines in Fig. 3 for **3c,d**. These polymers exhibited absorption tailing to 460 nm for **3c** and to 420 nm for **3d**. Similar tailing profiles were observed for other polymers **3a,b,e,f** as well. On the other hand, **3a–d** showed fluorescence emission spectra with multipeaks (Table 2). Most intense peaks for the *p*-phenylene polymers emerged in visible blue to green region (450–500 nm), while those of the corresponding *m*-phenylene polymers were observed in ultraviolet region (330–380 nm), as typically shown for **3c,d** (Fig. 3). The quantum yields of the emission for **3c,d** were estimated at 0.061 and 0.026, respectively, by the comparison with quinine sulfate as a standard. The main features of absorption and emission spectra for the *p*-phenylene polymers **3a,c** were similar to those obtained by Pt catalyst [5]. In addition to the solution state, spin-coated thin films of **3a–d** also exhibited blue to green light emission on exposure to UV light. Typically, the absorption and emission spectra of the thin films of **3c,d** were depicted as broken lines in Fig. 3. The absorption spectra of the thin films resembled those of the solutions for both **3c** and **3d**. The emission spectrum of the film of **3c** was also similar to that of the solution of **3c**, showing largest peaks around 460–500 nm. The film of **3d**, however, exhibited an emission spectrum with largely increased peaks around 420–450 nm, which was significantly different from that of the solution with the largest peaks around 340–360 nm. The visible light-emitting properties of the thin films may be applicable to some optoelectric devices, although futher studies will be required to know the mechanism of the light emission and the difference between **3c** and **3d**.

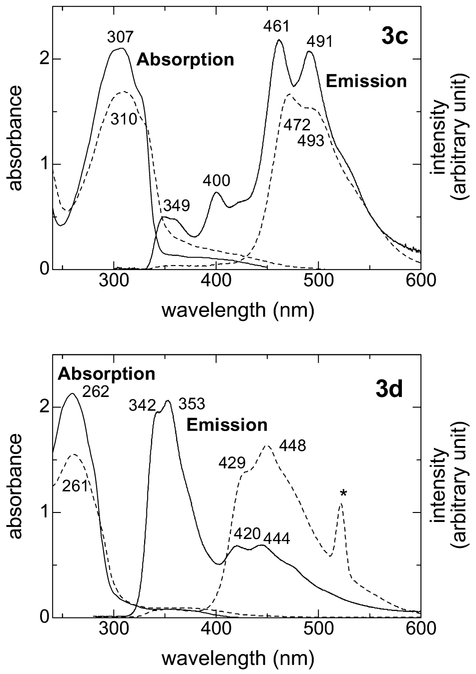


Fig. 3. UV/vis absorption and fluorescence emission spectra of **3c** (top) and **3d** (bottom); solid line for solution (6.67×10^{-5} M (mol monomer unit/l) in chloroform), broken line for thin film (ca. 100 nm, on quartz glass). *Second order diffraction peak.

In TGA **3a–f** showed rather high thermal stability. For **3a–d** with *p*- or *m*-phenylene linkage and Ph and/or Me substituents at the silicon atom, the 5% weight loss temperatures (Td_5) under nitrogen ranged from 436 to 461 °C, while the char yields at 980 °C were in the range of 56–62% (Table 2, Fig. 4). The difference in thermal stability between them were rather small, as observed for **3a,c** obtained by Pt catalyst [5]. In contrast with the above divinylene polymers, monovinylene polymers obtained by Pt-catalyzed hydrosilylation of *p*- or *m*-(dimethylsilyl)phenylacetylene seemed to have somewhat lower thermal stability; the Td_5 and the char yield at 800 °C were reported

Table 2
Photophysical and thermal properties of **3**

3	UV/vis absorption[a] λ_{max}, nm ($\varepsilon_{monomer\ unit}$)	Fluorescence emission[a] λ_{max}, nm (excitation,[b] nm)	TGA[c] Td_5, °C	Char yield,[d] %
3a	306 (2.9×10^4)	347, 400, 421, 460, 489 (270)	445	56
3b	259 (2.8×10^4)	341, 352, 421, 443 (256)	461	62
3c	307 (3.1×10^4)	349, 400, 461, 491 (267)	436	57
3d	262 (3.2×10^4)	342, 353, 420, 444 (256)	438	61
3e	304 (2.7×10^4)	361, 397, 458, 486[e] (335)	501	75
3f	262 (3.2×10^4)	337, 353[e] (291)	488	73

[a] In chloroform, $c = 6.67 \times 10^{-5}$ M (mol monomer unit/l).

[b] Excitation wavelength.

[c] Under nitrogen, 10 °C/min heating rate.

[d] At 980 °C.

[e] Emission peaks for **3e,f** were relatively small.

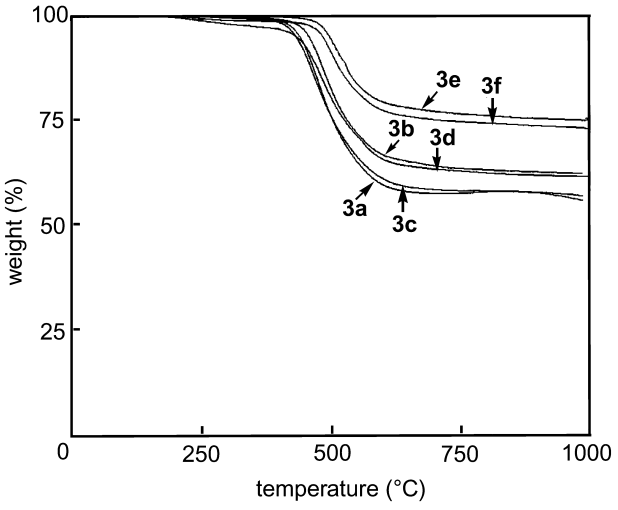


Fig. 4. TGA of **3a–f** (under nitrogen).

to be 378 °C and 65% for the former and 315 °C and 40% for the latter [4]. In the present divinylene polymers, introduction of a vinyl substituent at the silicon atom appeared to significantly improve thermal stability. Thus, the Td_5 values and the char yields were 501 °C and 75% for **3e** and 488 °C and 73% for **3f**. This might be due to occurrence of thermal crosslinking between the vinyl groups during the heating process of TGA. A phenylene-silylene polymer with a vinyl substituent at the silicon atom ($[p\text{-}C_6H_4\text{–SiMe}(CH{=}CH_2)]_n$) was also previously reported to have rather high thermal stability under nitrogen ($Td_5 = 486$ °C, char yield at 1000 °C = 71%) [18].

In summary, the Pd-PCy_3 system has proved to be an efficient catalyst for hydrosilylation polymerization of dihydrosilanes with diynes, providing silylene-divinylene polymers with various substituents including a vinyl group. Application of the catalysis to preparation of new functional materials will be the coming subject.

3. Experimental

3.1. Materials

Dihydrosilanes **1a,b** and diynes **2a,b** were commercially available, and used after purification by distillation in the presence of CaH_2 under nitrogen or by sublimation. Vinylsilane **1c** was synthesized by a literature method [19]. Benzene and benzene-d_6 were dried with CaH_2 and distilled under nitrogen. $Pd_2(dba)_3$, $PdCl_2(PCy_3)_2$, and PCy_3 were purchased and used as they were.

3.2. Instruments

^1H and ^{29}Si NMR spectra were measured by a Bruker ARX-300 instrument. IR spectra were recorded on a JASCO FT/IR-5000 spectrometer. UV/vis absorption spectra were collected with Shimadzu UV-3100 and Hitachi U-3010 spectrometers, while fluorescence emission spectra were recorded on Hitachi F-4500 and JASCO FP-6500 spectrometers. The fluorescence quantum yields of polymer samples (in THF, $c = 2.0 \times 10^{-6}$ mol monomer unit/l) were estimated according to the reported method using quinine sulfate as a standard ($\Phi = 0.55$) [20]. The absorption and emission spectra of a polymer in solid state were measured using a thin film prepared by spin-coating of a toluene solution of the polymer on a quartz glass plate. TGA measurements were conducted using a Seiko TG/DTA 320 instrument. Molecular weights of polymers were estimated by a GPC system equipped with a high pressure Shimadzu 10ADp pump, Polymer Laboratories PLgel MIXED-B columns, and a Shimadzu RID-10A detector using polystyrene standards and toluene eluent.

3.3. Polymerization reactions of **1** with **2**

Typically, to a mixture of **1a** (0.40 mmol), **2a** (0.40 mmol), and benzene-d_6 (0.28 ml) in a NMR tube was added $Pd_2(dba)_3$-PCy_3 benzene-d_6 solution (0.0024 mmol Pd, P/Pd = 2, 0.12 ml) under nitrogen, and the NMR tube was sealed. The mixture was heated at 70 °C, and the progress of the reaction was monitored by NMR. After heating for 0.5 h acetylenic proton signals (2.7–2.9 ppm) disappeared almost completely, while small signals still remained in Si–H region (around 5.0 ppm). Purification by precipitation from benzene–2-propanol gave analytically pure **3a** as pale yellow powder (0.345 mmol, 86% yield). Separate reaction using benzene in place of benzene-d_6 in a glass tube also gave a similar result. Similarly, polymers **3b–f** were obtained using the $Pd_2(dba)_3$-PCy_3 or $PdCl_2(PCy_3)_2$ catalyst system.

Spectral and analytical data for **3a–f** are as follows.

3a [5]: ^1H NMR (C_6D_6) δ 0.36–0.77 (br m, Si–CH_3), 5.05–5.12 (br m, terminal Si–H), 5.70–5.84 and 6.07–6.21 (each br m, Si–C=CH_2), 6.63–6.87 (br doublet-like, J = ca. 19 Hz, Si–CH=C), 7.00–7.90 (br m, aromatic H and Si–C=CH); ^{29}Si NMR (C_6D_6) δ − 15.7, − 15.6, − 13.4; IR (KBr) 2124 (ν_{Si-H}), 1603, 1508, 1429, 1253, 1110, 986, 803, 737, 698 cm^{-1}. Anal. calcd for $(C_{17}H_{16}Si)_n$: C, 82.20; H, 6.49. Found: C, 81.88; H, 6.36.

3b: ^1H NMR (C_6D_6) δ 0.30–0.74 (br m, Si–CH_3), 5.01–5.11 (br m, terminal Si–H), 5.65–5.85 and 6.00–6.20 (each br m, Si–C=CH_2), 6.50–6.80 (br doublet-like, J = ca. 19 Hz, Si–CH=C), 6.80–7.90 (br m, aromatic H and Si–C=CH); ^{29}Si NMR (C_6D_6) δ − 15.7, − 15.6, − 13.6; IR (KBr) 2120 (ν_{Si-H}), 1605, 1570, 1429, 1251, 1191, 1110, 988, 828, 789, 735, 696 cm^{-1}. Anal. calcd for $(C_{17}H_{16}Si)_n$: C, 82.20; H, 6.49. Found: C, 81.87; H, 6.53.

3c [5]: ^1H NMR (C_6D_6) δ 5.67–5.85 (br m, terminal Si–H and Si–C=CH^aH^b), 6.08–6.32 (br m, Si–C=CH^aH^b), 6.70–7.04 (br doublet-like, J = ca. 19 Hz, Si–CH=C), 7.05–7.85 (br m, aromatic H and Si–C=CH); ^{29}Si NMR (C_6D_6) δ − 18.4, − 18.3, − 15.9; IR (KBr) 2123 (ν_{Si-H}), 1595, 1429, 1108, 988, 806, 779, 737, 698 cm^{-1}. Anal. calcd for $(\mathrm{C}_{22}\mathrm{H}_{18}\mathrm{Si})_n$: C, 85.12; H, 5.84. Found: C, 84.66; H, 5.82.

3d: ^{1}H NMR ($\mathrm{C}_6\mathrm{D}_6$) δ 5.67–5.84 (br m, terminal Si–H and Si–C=CH^{a}H$^{\mathrm{b}}$), 6.08–6.32 (br m, Si–C=CH$^{\mathrm{a}}$$H^{\mathrm{b}}$), 6.70–7.02 (br doublet-like, J = ca. 19 Hz, Si–CH=C), 7.02–7.87 (br m, aromatic H and Si–C=CH); ^{29}Si NMR ($\mathrm{C}_6\mathrm{D}_6$) δ −18.4, −18.3, −16.2; IR (KBr) 2125 ($\nu_{\mathrm{Si-H}}$), 1570, 1429, 1191, 1108, 992, 804, 766, 737, 698 cm^{-1}. Anal. calcd for $(\mathrm{C}_{22}\mathrm{H}_{18}\mathrm{Si})_n$: C, 85.12; H, 5.84. Found: C, 84.45; H, 5.86.

3e: ^{1}H NMR ($\mathrm{C}_6\mathrm{D}_6$) δ 5.10–5.30 (br m, terminal Si–H), 5.70–6.33 and 6.40–6.61 (each br m, Si–CH=CH_2 and C=CH_2), 6.61–6.89 (br doublet-like, J = ca. 19 Hz, Si–CH=C–C), 6.97–7.90 (br m, aromatic H and Si–C=CH–C); ^{29}Si NMR ($\mathrm{C}_6\mathrm{D}_6$) δ −21.7, −21.6, −19.5; IR (KBr) 2120 ($\nu_{\mathrm{Si-H}}$), 1591, 1506, 1429, 1404, 1191, 1110, 988, 961, 804, 777, 737, 698 cm^{-1}. Anal. calcd for $(\mathrm{C}_{18}\mathrm{H}_{16}\mathrm{Si})_n$: C, 83.02; H, 6.19. Found: C, 82.98; H, 6.29.

3f: ^{1}H NMR ($\mathrm{C}_6\mathrm{D}_6$) δ 5.14–5.28 (br m, terminal Si–H), 5.65–6.31 and 6.35–6.62 (each br m, Si–CH=CH_2 and C=CH_2), 6.62–6.90 (br doublet-like, J = ca. 19 Hz, Si–CH=C–C), 6.90–7.90 (br m, aromatic H and Si–C=CH–C); ^{29}Si NMR ($\mathrm{C}_6\mathrm{D}_6$) δ −21.8, −21.6, −19.8; IR (KBr) 2126 ($\nu_{\mathrm{Si-H}}$), 1589, 1570, 1429, 1402, 1193, 1110, 990, 959, 803, 766, 737, 698 cm^{-1}. Anal. calcd for $(\mathrm{C}_{18}\mathrm{H}_{16}\mathrm{Si})_n$: C, 83.02; H, 6.19. Found: C, 82.84; H, 6.28.

*3.4. A model reaction of **1a** with **4***

To a mixture of **1a** (0.20 mmol), **4** (0.40 mmol), and benzene-d_6 (0.03 ml) in a NMR tube was added $\mathrm{Pd}_2(\mathrm{dba})_3$-$\mathrm{PCy}_3$ benzene-d_6 solution (0.0024 mmol Pd, P/Pd = 2, 0.12 ml) under nitrogen, and the NMR tube was sealed. When the mixture was heated at 70 °C for 2 h, PhMeSi(CH=CHPh)$_2$ (**5ββ**, *trans,trans* isomer) and PhMeSi(CH=CHPh)[C(=CH_2)Ph] (**5βα**, *trans* isomer) were indicated to be formed in ≥90% combined yield with the ratio of **5ββ**:**5βα** being 80:20. Purification by preparative TLC (hexane:triethylamine:ethyl acetate = 50:1:1) gave the products as a colorless oil (a mixture of **5ββ** and **5βα**, totally 0.12 mmol, 60% yield). ^{1}H NMR ($\mathrm{C}_6\mathrm{D}_6$) δ 0.55 and 0.57 (each s, Si–CH_3, **5βα** and **5ββ**, respectively), 5.77 and 6.11 (each d, each J = 2.8 Hz, Si–C=CH_2, **5βα**), 6.71 (d, J = 19.1 Hz, Si–CH=C, **5ββ**), 6.72 (d, J = 19.1 Hz, Si–CH=C, **5βα**), 6.97–7.37 and 7.63–7.74 (each m, aromatic H and Si–C=CH–C, **5ββ** and **5βα**); ^{29}Si NMR ($\mathrm{C}_6\mathrm{D}_6$) δ −15.7 (**5ββ**), −13.5 (**5βα**); IR (neat) 1603, 1574, 1495, 1448, 1429, 1251, 1112, 992, 841, 795, 733, 698, 690 cm^{-1}. Anal. calcd for $\mathrm{C}_{23}\mathrm{H}_{22}\mathrm{Si}$: C, 84.61; H, 6.79. Found: C, 84.61; H, 6.68.

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