Synthesis and mass spectrometric study of α,ω-diallylpermethyloligosilanes

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Representatives of the homologous series of α,ω-diallylpermethyloligosilanes were synthesized for the first time by the reaction of α,ω-dichloropermethyloligosilanes $Cl(Me_2Si)_nCl$ (n = 2—6) with allylmagnesium chloride. Fragmentation of α,ω-diallylpermethyloligosilanes under electron impact ionization was studied by mass spectrometry.

Key words: α,ω-dichloropermethyloligosilanes, allylmagnesium chloride, α,ω-diallylpermethyloligosilanes, NMR spectra, IR spectra, UV spectra, mass spectra.

α,ω-Diallylpermethyloligosilanes are used as the starting compounds for the synthesis of polycarbosilanes, which are of considerable practical importance as ceramic-forming polymers.[1] However, data on the preparation of such oligomers are scarce. Only the syntheses of 1,2-diallyltetramethyldisilane by thermal isomerization of 1,2-cyclopropyltetramethyldisilane[2] (7.0% yield), cocondensation of allyldimethylchlorosilane and vinyldimethylchlorosilane with magnesium[3] (2.4% yield), and the reaction of allylmagnesium bromide with 1,2-dichlorotetramethyldisilane[4] (the yield was not reported) were described.

In the present study, we synthesized representatives of the homologous series of α,ω-diallylpermethyloligosilanes $All(Me_2Si)_nAll$ (**2a—e**) by the reaction of α,ω-dichloropermethyloligosilanes $Cl(Me_2Si)_nCl$ (**1a—e**) with allylmagnesium chloride, examined the spectroscopic characteristics of the resulting oligomers, and studied their fragmentation under electron impact (EI) ionization.

Results and Discussion

The reaction was carried out in Et_2O at the boiling temperature of the solvent. The yields of oligomers **2a—e** were 50—75% (Scheme 1).

Scheme 1

$$\underset{\textbf{1a—e}}{Cl(Me_2Si)_nCl} + AllMgCl \longrightarrow \underset{\textbf{2a—e}}{All(Me_2Si)_nAll}$$

n = 2 (**a**), 3 (**b**), 4 (**c**), 5 (**d**), 6 (**e**)

As distinct from other nonfunctional and bifunctional permethylhexasilanes $X(Me_2Si)_6X$ (X = Me, Ph, Cl, OH, *etc.*), which were prepared in the crystalline state, diallylhexasilane **2e**, like diallyloligosilanes **2a—d**, is a colorless transparent liquid.

The structures of oligomers **2a—e** were studied by ^1H and ^{29}Si NMR, IR, UV, and Raman spectroscopy. In the ^{29}Si NMR spectra of oligomers **2a—e**, the signals for the Si_a, Si_b, and Si_c atoms are shifted downfield as the number of sequentially linked Si atoms increases (Table 1).

The IR and Raman spectra of oligomers **2a—e** (see Table 1) show intense lines at 1630 cm^{-1} characteristic of C=C stretching vibrations of allylsilanes.[5] In addition, the Raman spectra have lines corresponding to Si—Si stretching vibrations of oligosilanes in regions of 360—400 cm^{-1} (symmetric vibrations) and 440—500 cm^{-1} (antisymmetric vibrations).[6]

Oligomers **2a—e** absorb in the UV region. The position of the absorption maximum λ_{max} depends on the number of the $SiMe_2$ groups in oligosilanes **2a—e**, and λ_{max} is shifted bathochromically as the number of these groups increases (see Table 1).

Mass spectrometric study of diallyloligosilanes 2a—e. The fragmentation of oligomers **2a—e** and, for comparison, of diallyldimethylsilane All_2SiMe_2 (**3**) under electron impact (EI) ionization was studied by GLC-mass spectrometry. Silane **3** is formally the first member of the homologous series of diallyloligosilanes, which differ by one $SiMe_2$ unit, but it contains no Si—Si bonds. The fragmentation of silane **3**, analogously to fragmentation of other silanes,[7] occurs with elimination of the Me and All groups from the Si atom (Table 2). In the mass spectra of oligomers **2a—e**, in contrast to the mass spectrum of silane **3**, the intensity of the molecular ion peak $[M]^+$ is low (see Table 2), which is typical also of certain mono- and bifunctional linear permethyloligosilanes. For ex-

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Table 1. Selected spectroscopic characteristics of oligomers **2a—e**

Compound[a]	^{29}Si NMR, δ			IR,[b] ν/cm^{-1} C=C	Raman spectrum,[b] Δν/cm^{-1}		UV spectrum,[c] λ_{max}/nm
	Si_a	Si_b	Si_c		C=C	Si—Si	
$AllSi_aSiAll$ (**2a**)	−17.45	—	—	1634	1632	400	223
$AllSi_aSi_bSiAll$ (**2b**)	−14.57	−47.17	—	1627	1630	380, 457	230
$AllSi_aSi_bSiSiAll$ (**2c**)	−14.19	−43.94	—	1634	1630	368, 473	239
$AllSi_aSi_bSi_cSiSiAll$ (**2d**)	−13.88	−42.20	−40.12	1627	1630	362, 442, 478	254
$AllSi_aSi_bSi_cSiSiSiAll$ (**2e**)	−13.80	−42.02	−38.59	1634	1632	360, 478	262

[a] The Me groups at the Si atoms are omitted.
[b] The complete spectra are given in the Experimental section.
[c] Without a solvent.

Table 2. Mass spectra of diallyloligosilanes **2a—e** and compound **3**

Ion	*m/z* (I_{rel} (%))					
	2a	**2b**	**2c**	**2d**	**2e**	**3**
$[M]^+$	198 (1.3)	256 (1.1)	314 (1.8)	372 (1.1)	430 (0.9)	140 (3.5)
$[M - Me]^+$	183 (0.8)	241 (0.9)	299 (0.8)	357 (0.2)	415 (0.0)	125 (19.2)
$[M - All]^+$	157 (100.0)	215 (71.1)	273 (6.2)	331 (1.1)	389 (0.6)	99 (100.0)
$[M - Me - C_2H_4]^+$	155 (12.9)	213 (1.8)	—	—	—	97 (61.6)
$[M - All - C_2H_2]^+$	131 (17.2)	189 (1.6)	—	—	—	73 (38.1)
$[M - All - C_2H_4]^+$	129 (83.5)	187 (12.3)	—	—	—	71 (48.9)
$[M - All - C_3H_4]^+$	117 (30.6)	175 (1.4)	—	—	—	59 (47.1)
A	99 (20.7)	99 (27.0)	99 (23.0)	99 (16.1)	99 (21.1)	—
B	—	157 (44.8)	157 (79.0)	157 (35.6)	157 (32.8)	—
C	—	—	215 (77.5)	215 (40.4)	215 (97.9)	—
D	—	—	—	273 (0.5)	273 (8.1)	—
E	—	—	—	—	331 (0.7)	—
$[A - CH_4]^+$	83 (15.2)	83 (7.3)	83 (7.2)	83 (5.9)	83 (8.7)	—
$[A - C_2H_2]^+$	73 (92.9)	73 (81.2)	73 (100.0)	73 (64.9)	73 (85.7)	—
$[A - C_2H_4]^+$	71 (13.9)	71 (5.2)	71 (5.4)	71.0 (8.0)	71 (8.7)	—
$[B - CH_4]^+$	—	141 (100.0)	141 (99.6)	141 (100.0)	141 (100.0)	—
$[B - C_2H_2]^+$	—	131 (43.7)	131 (63.9)	131 (36.3)	131 (37.6)	—
$[B - C_2H_4]^+$	—	129 (46.4)	129 (53.3)	129 (36.8)	129 (36.2)	—
$[B - C_2H_6]^+$	—	127 (72.9)	127 (43.5)	127 (35.5)	127 (41.9)	—
$[B - C_3H_4]^+$	—	117 (30.6)	117 (41.0)	117 (29.4)	117 (28.9)	—
$[C - CH_4]^+$	—	—	199 (78.7)	199 (45.6)	199 (55.9)	—
$[C - C_2H_6]^+$	—	—	185 (54.1)	185 (29.1)	185 (34.2)	—
$[C - C_3H_4]^+$	—	—	175 (8.1)	175 (6.7)	175 (16.8)	—
$[C - C_3H_6]^+$	—	—	173 (14.6)	173 (11.9)	173 (19.5)	—
$[D - CH_4]^+$	—	—	—	257 (7.2)	257 (11.3)	—
$[D - C_2H_6]^+$	—	—	—	243 (8.7)	243 (11.3)	—
$[E - CH_4]^+$	—	—	—	—	315 (9.3)	—
$[E - C_3H_6]^+$	—	—	—	—	289 (6.1)	—

ample, the intensity of the $[M]^+$ peak in the mass spectra of halogen-containing oligosilanes,[8,9] *viz.*, 1-chloropermethyloligosilanes $Cl(SiMe_2)_nMe$ (n = 2—4 and 6) and α,ω-dihalopermethyloligosilanes $Hal(SiMe_2)_nHal$ (Hal = Cl, Br, or I; n = 2—6), is 0.1—2.8%. By contrast, the $[M]^+$ peaks in the mass spectra of permethyloligosilanes[10] $Me(SiMe_2)_nMe$ (n = 3—5) have a higher intensity (15—21%).

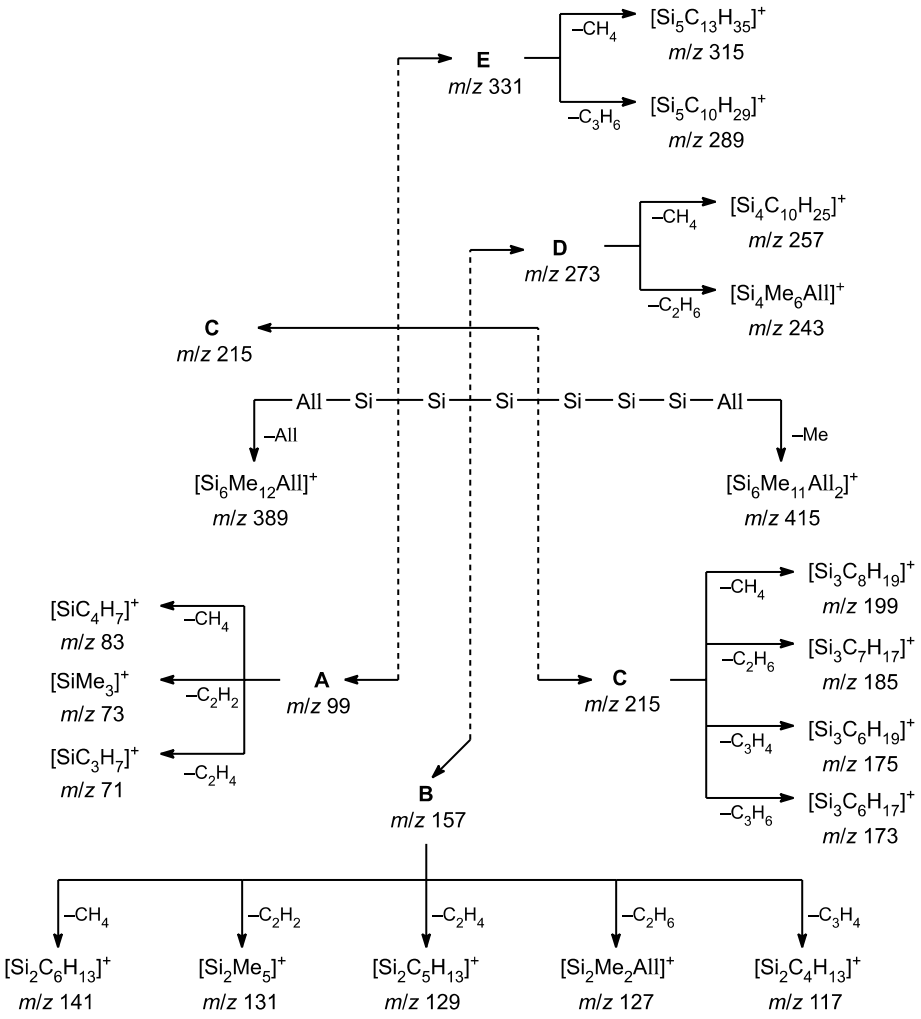
For oligomers **2a—e**, the main fragmentation pathways of the $[M]^+$ ion involve elimination of the Me and All groups from the Si atom and the Si—Si bond cleavage. The intensities of the ion peaks $[M - Me]^+$ in the mass spectra of oligomers **2a—e**, unlike those in the spectra of simple silanes and compound **3**, are low and decrease virtually to zero in going from oligomer **2a** to **2e** (*i.e.*, as the number of $SiMe_2$ units increases). The allyl group is

eliminated more readily than the methyl group, and the ion peaks $[M - All]^+$ in the mass spectra of disilane **2a** and trisilane **2b** have a high intensity. However, in this case an increase in the number of $SiMe_2$ units to four—six (oligomers **2c—e**) also leads to a sharp decrease in the intensity of these ion peaks (see Table 2). Suppression of elimination of the substituents at the Si atom with increasing chain length is common to linear permethyloligosilanes[10] and halooligosilanes,[8,9] which is associated with a lower energy of the Si—Si bond compared to that of the Si—C bond and, consequently, with the occurrence of alternative fragmentation pathways. Actually, the mass spectra of oligomers **2a—e** show intense ion peaks corresponding to the Si—Si bond cleavage. For example, fragmentation of the $[M]^+$ ion of oligomer **2e** gives rise to the ions **A**, **B**, **C**, **D**, and **E** of the $[All - (SiMe_2)_n]^+$ type (n = 1—5, respectively), which differ from each other by the homologous $SiMe_2$ unit (Scheme 2). In the mass spectra of oligomers **2a—e**, the peaks of the ions **A**, **B**, and **C** containing one, two, and three Si atoms, respectively, are most intense (16—98%), whereas the intensity of the peaks of the ions **D** and **E** with n = 4 or 5 is low (0.5—8%) (see Table 2).

In the case of other linear oligosilanes, the formation of the $[X(SiMe_2)_n]^+$ ions (X = Me or Hal) is also the major process.[8—10] For example, the intensities of the corresponding ion peaks at *m/z* 189, 131, and 73 in the spectrum of decamethyltetrasilane[10] are 34.3, 53.0, and 100%, respectively.

Presumably, the ions **A**—**E** can be formed both through the direct cleavage of the Si—Si bonds in the molecular ion and through the successive fragmentation of the ions **E** → **D** → **C** → **B** → **A** with elimination of the silylene group Me_2Si. In addition, the fragment ions corresponding to elimination of the substituents at the Si atom, *viz.*, $[M - Me]^+$ and $[M - All]^+$, can serve as a source of the ions **A**—**E**. For permethyloligosilanes[10] and α,ω-dihalopermethyloligosilanes,[8,9] similar fragmentation processes

Scheme 2



were confirmed by the results of studies of metastable transitions. Elimination of silylene Cl_2Si from the molecular and fragment ions was observed also in the course of fragmentation of $[M]^+$ of α,ω-dihydridoperchlorooligosilanes.[11]

Further fragmentation of the fragment ions $[M - Me]^+$, $[M - All]^+$, **A—E**, compound **3**, and diallyloligosilanes **2a—e** is accompanied to a greater or lesser extent by elimination of hydrocarbon molecules (CH_4, C_2H_2, C_2H_4, C_2H_6, C_3H_4, and C_3H_6) in accordance with the parity rule (see Table 2). It can be hypothesized that these fragmentation pathways involve decomposition of the allyl group, because elimination of hydrocarbon molecules from fragment ions of α,ω-dihalooligosilanes[8,9] and permethyloligosilanes[10] was not observed. By contrast, the fragment ions in the mass spectra of 1,1,2,2,3,3-hexaethyltrisilane and 1-triethylsilylhexaethyltrisilane[12] are characterized by the fragmentation with successive elimination of ethylene and ethane molecules.

Therefore, the main fragmentation pathway of oligomers **2a—e** under EI ionization involves elimination of the methyl and allyl substituents at the Si atom and the Si—Si bond cleavage, the intensities of the resulting ions being determined by the number of $SiMe_2$ units in the oligomer molecules.

Experimental

The ^1H and ^{29}Si NMR spectra were recorded on a Bruker WP-400 SY spectrometer in $CDCl_3$ with Me_4Si as the internal standard. The IR spectra were measured in a 400—3700-cm^{-1} region on a Specord M-82 spectrophotometer in a thin layer between KBr glasses. The UV spectra were recorded in a 200—335-nm region on a Specord M-40 spectrophotometer. The Raman spectra were measured in a 200—4000-cm^{-1} region on a T-64000 spectrometer (Jobin Yvon) equipped with a CCD detector (the exciting wavelength was 514.5 nm). The GLC-mass spectrometric analysis was carried out on an HP-5890 instrument (20 m × 0.32-mm capillary column, SE-30 liquid phase, helium as carrier gas, the temperature of the injector and transition lines was 250 °C, ionizing voltage was 70 eV, temperature programming from 60 to 300 °C at a rate of 7 K min^{-1}).

Dichlorooligosilanes **1a—e** were synthesized according to a procedure described earlier.[13] Diallyldimethylsilane (**3**) was synthesized according to a known procedure (86% yield).[4] Diethyl ether was dehydrated by refluxing followed by distillation under a stream of N_2 over metallic sodium in the presence of benzophenone. All reactions were carried out under dry argon.

1,2-Diallyltetramethyldisilane (2a). A solution of dichlorodisilane **1a** (5.0 g, 26.7 mmol) in anhydrous Et_2O (10 mL) was added dropwise with vigorous stirring to a suspension of AllMgCl, which was prepared from Mg (2.84 g, 117 mmol) and AllCl (8.19 g, 107 mmol), in anhydrous Et_2O (30 mL). The reaction mixture was brought to reflux and stirred for 10 h. Then a saturated NH_4Cl solution was added. The organic layer was separated, washed with water to neutral pH, and dried with Na_2SO_4. The diethyl ether was distilled off and the residue was fractionated. The yield of diallyldisilane **2a** was 3.17 g (60.0%), b.p. 76—78 °C (10 Torr). Found (%): C, 60.87; H, 11.28; Si, 27.85. $C_{10}H_{22}Si_2$. Calculated (%): C, 60.52; H, 11.17; Si, 28.31. ^1H NMR, δ: 0.18 (s, 12 H, 4 Me); 1.72 (d, 4 H, 2 CH_2, J = 8.1 Hz); 4.95 (d, 4 H, 2 $=CH_2$, J = 14.5 Hz); 5.83—5.94 (m, 2 H, 2 CH). IR, ν/cm^{-1}: 3077 (ν(=CH)); 2953, 2891 (ν(CH)); 1634 (ν(C=C)); 1425, 1253 (δ(CH_3)); 1154 (δ(SiCH)); 895 (ρ($=CH_2$)); 830, 796, 646 (ν(SiC)). Raman spectrum, Δν/cm^{-1}: 3082 (ν(=CH)); 2954, 2893 (ν(CH)); 1632 (ν(C=C)); 1424, 1392, 1299 (ρ(CH_3)); 1150 (δ(SiCH)); 990 (ρ($CH=CH_2$)); 931 (ρ($=CH_2$)); 696, 657, 582 (ν(SiC)); 410 (δ(CC=C)); 400 (ν(SiSi)).

1,3-Diallylhexamethyltrisilane (2b). Diallyltrisilane **2b** was prepared according to the above-described procedure from Mg (2.18 g, 89.7 mmol), AllCl (6.24 g, 81.6 mmol), and dichlorotrisilane **1b** (5.0 g, 20.4 mmol) in a yield of 3.4 g (65.1%), b.p. 45—47 °C (0.5 Torr). Found (%): C, 56.45; H, 11.27; Si, 32.28. $C_{12}H_{28}Si_3$. Calculated (%): C, 56.17; H, 11.00; Si, 32.83. ^1H NMR, δ: 0.30 (s, 12 H, 4 Me); 0.35 (s, 6 H, 2 Me); 1.84 (d, 4 H, 2 CH_2, J = 8.4 Hz); 5.02 (d, 4 H, 2 $=CH_2$, J = 13.4 Hz); 5.94—6.07 (m, 2 H, 2 CH). IR, ν/cm^{-1}: 3080 (ν(=CH)); 2950, 2891 (ν(CH)); 1627 (ν(C=C)); 1400, 1248 (δ(CH_3)); 1151 (δ(SiCH)); 897 (ρ($=CH_2$)); 832, 789, 724, 649 (ν(SiC)). Raman spectrum, Δν/cm^{-1}: 3080 (ν(=CH)); 2953, 2903 (ν(CH)); 1630 (ν(C=C)); 1418, 1395, 1299, 1240, 1194 (ρ(CH_3)); 1152 (δ(SiCH)); 990 (ρ($CH=CH_2$)); 931 (ρ($=CH_2$)); 725, 687, 658, 638 (ν(SiC)); 457, 380 (ν(SiSi)); 403 (δ(CC=C)).

1,4-Diallyloctamethyltetrasilane (2c). Analogously to the synthesis of diallyldisilane **2a**, diallyltetrasilane **2c** was prepared from Mg (1.07 g, 44 mmol), AllCl (3.06 g, 40 mmol), and dichlorotetrasilane **1c** (3.03 g, 10 mmol) in a yield of 2.0 g (63.7%), b.p. 81—83 °C (0.5 Torr). Found (%): C, 53.74; H, 11.02; Si, 35.24. $C_{14}H_{34}Si_4$. Calculated (%): C, 53.42; H, 10.89; Si, 35.69. ^1H NMR, δ: 0.05 and 0.20 (both s, 12 H each, 8 Me); 1.58 (d, 4 H, 2 CH_2, J = 8.8 Hz); 4.77 (d, 4 H, 2 $=CH_2$, J = 14.5 Hz); 5.50—5.85 (m, 2 H, 2 CH). IR, ν/cm^{-1}: 3077 (ν(=CH)); 2953, 2891 (ν(CH)); 1634 (ν(C=C)); 1402, 1249 (δ(CH_3)); 1153 (δ(SiCH)); 892 (ν($=CH_2$)); 836, 779, 734, 643 (ν(SiC)). Raman spectrum, Δν/cm^{-1}: 3081 (ν(=CH)); 2954, 2896 (ν(CH)); 1630 (ν(C=C)); 1415, 1398, 1299, 1241 (ρ(CH_3)); 1152 (δ(SiCH)); 894 (ν($=CH_2$)); 743, 726, 685, 660 (ν(SiC)); 473, 368 (ν(SiSi)); 409 (δ(CC=C)).

1,5-Diallyldecamethylpentasilane (2d). Analogously to the synthesis of diallyldisilane **2a**, diallylpentasilane **2d** was prepared from Mg (0.89 g, 36.5 mmol), AllCl (2.54 g, 33.2 mmol), and dichloropentasilane **1c** (3.0 g, 8.3 mmol) in a yield of 2.3 g (74.4%), b.p. 99—101 °C (0.4 Torr). Found (%): C, 51.82; H, 10.95; Si, 37.23. $C_{16}H_{40}Si_5$. Calculated (%): C, 51.53; H, 10.81; Si, 37.66. ^1H NMR, δ: 0.25 and 0.33 (both s, 12 H each, 8 Me); 0.38 (s, 6 H, 2 Me); 1.78 (d, 4 H, 2 CH_2, J = 8.1 Hz); 5.03 (d, 4 H, 2 $=CH_2$, J = 13.5 Hz); 5.70—6.15 (m, 2 H, 2 CH). IR, ν/cm^{-1}: 3080 (ν(=CH)); 2945, 2891 (ν(CH)); 1627 (ν(C=C)); 1405, 1248 (δ(CH_3)); 1151 (δ(SiCH)); 897 (ν($=CH_2$)); 832, 773, 730, 692, 643 (ν(SiC)). Raman spectrum, Δν/cm^{-1}: 3080 (ν(=CH)); 2952, 2897 (ν(CH)); 1630 (ν(C=C)); 1417, 1398, 1299, 1238, 849 (ρ(CH_3)); 1151 (δ(SiCH)); 892 (ν($=CH_2$)); 743, 725, 684, 659, 642 (ν(SiC)); 478, 442, 362 (ν(SiSi)); 408 (δ(CC=C)).

1,6-Diallyldodecamethylhexasilane (2e). Analogously to the synthesis of diallyldisilane **2a**, diallylhexasilane **2e** was prepared from Mg (0.64 g, 26.3 mmol), AllCl (1.84 g, 24.1 mmol), and

dichlorohexasilane **1e** (2.52 g, 6.0 mmol) in a yield of 1.3 g (50.4%), b.p. 133—137 °C (0.5 Torr). Found (%): C, 50.40; H, 10.91; Si, 38.69. $C_{18}H_{46}Si_6$. Calculated (%): C, 50.15; H, 10.76; Si, 39.09. ^{1}H NMR, δ: 0.28, 0.36, and 0.39 (all s, 12 H each, 12 Me); 1.81 (d, 4 H, 2 CH_2, J = 8.1 Hz); 5.02 (d, 4 H, 2 $=CH_2$, J = 13.7 Hz); 5.91—6.01 (m, 2 H, 2 CH). IR, ν/cm^{-1}: 3077 (ν(=CH)); 2953, 2891 (ν(CH)); 1634 (ν(C=C)); 1402, 1249 (δ(CH_3)); 1153 (δ(SiCH)); 892 (ν($=CH_2$)); 836, 796, 768, 734, 689, 655 (ν(SiC)). Raman spectrum, Δν/cm^{-1}: 3080 (ν(=CH)); 2958, 2893 (ν(CH)); 1632 (ν(C=C)); 1400, 1300, 1253 (ρ(CH_3)); 1150 (δ(SiCH)); 725, 682, 660 (ν(SiC)); 478, 360 (ν(SiSi)); 410 (δ(CC=C)).

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