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Journal of Organometallic Chemistry 611 (2000) 26–31

Journal of Organo metallic Chemistry

Electroreductive synthesis of oligosilanes and polysilanes with ordered sequences

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Received 21 February 2000; received in revised form 25 April 2000; accepted 10 May 2000

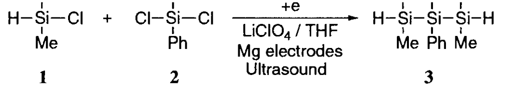
Abstract

The stepwise elongation of Si–Si or Si–Ge chain was achieved by the electroreductive cross-coupling reaction of chlorohydrosilanes with dichlorooligosilanes using magnesium electrodes. The electroreductive cross-coupling reaction of chlorodimethylsilane (**1**) with dichlorodiphenylsilane (**2**) or dichlorodiphenylgermane (**4**), for instance, gave 1,3-dihydro-1,1,3,3-tetramethyl-2,2-diphenyltrisilane (**3**) or bis(hydrodimethylsilyl)germane (**5**) in good yield. Compounds **3** and **5** were readily transformed to the corresponding 1,3-dichlorotrisilane (**6**) and bis(chlorosilyl)germane (**7**), respectively. The electroreductive polymerization of the resulting dichlorooligosilanes using magnesium electrodes in tetrahydrofuran was successfully applied to the synthesis of sequence-ordered polysilanes.

Keywords: 1,3-Dihydrotrisilanes; 1,3-Dichlorotrisilanes; Polysilanes; Silane–germane copolymers; Electroreductive polymerization; Magnesium electrode

1. Introduction

Polysilanes and polygermanes have been attracting considerable attention due to their potential in the preparation of new types of material showing conducting, photoconducting, and nonlinear optical properties [1]. They have usually been prepared by the Wurts type coupling reaction of organodichlorosilanes with alkali metals (the Kipping's method) [2], however, this method requires rather drastic reaction conditions, and hence, has a disadvantage in controlling the unit-structure. Several novel methods have opened new approaches to the synthesis of structure-ordered polysilanes, which are essential to the further development in the polysilane chemistry [3a]. The anionic polymerization of masked disilenes has provided a useful method for preparing some poly(disilanylene)s whose units are ordered in two sequences [3b,c]. The anionic ring-opening polymerization of cyclic oligosilanes is another method for obtaining structure-controlled polysilanes [4].



Scheme 1.

Recently we have been studying the synthesis of polysilanes by using electrochemical methods and it has been reported in previous papers [5] that the electroreduction of organodichlorosilanes with Mg electrodes led to the formation of polysilanes having monomodal and sharp molecular weight distribution.

In this paper we wish to report that our electroreduction system was successfully applied to the stepwise elongation of Si–Si chain and the synthesis of structure-controlled polysilanes.

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PII: S0022-328X(00)00327-2

Table 1
Electroreductive cross-coupling of chlorosilane **1** with dichlorosilane **2** [a]

Entry	Chlorosilane **1** (mol l^{-1})	Dichlorosilane **2** (mol l^{-1})	Electricity (F mol^{-1})	Yield of **3** [b] (%)
1	1.69	0.34	6	41
2	2.33	0.33	6	63
3	2.31	0.33	4	45
4	3.30	0.66	6	43
5	2.93	1.00	6	3
6 [c]	2.30	0.33	6	75
7 [c]	2.30	0.33	4	36

[a] The electroreduction was carried out in a single-compartment cell using Mg electrodes under sonication (47 kHz), and the polarity of electrodes was alternated with An interval of 15 s.

[b] Isolated.

[c] The solution of **1** and **2** was added dropwise during the electroreduction.

2. Results and discussion

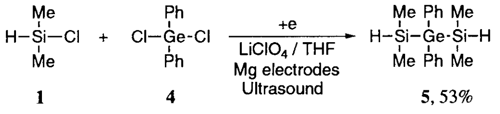
2.1. Electroreductive cross-coupling reaction of chlorodimethylsilane 1 with dichlorosilanes 2

We have already reported that the use of Mg electrodes is highly effective for the electroreductive formation of Si–Si bonds under mild reaction conditions [5]. The mildness of the reaction conditions of this electroreductive method is favorable for the synthesis of polysilanes having various functionalities such as Si–H bonds, which are known to be reactive under radical or anionic conditions [6]. The electroreductive cross-coupling reaction of chlorodimethylsilane (**1**) with dichlorodiphenylsilane (**2**), in fact, gave the corresponding trisilane **3** (Scheme 1). Some examples of the optimization of reaction conditions are summarized in Table 1.

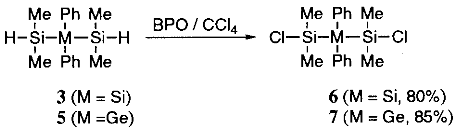
The electroreduction was carried out under constant current conditions in a single-compartment cell equipped with a Mg cathode and anode using $LiClO_4$ as a supporting electrolyte and dry tetrahydrofuran (THF) as a solvent [5]. During the electroreduction the electrolysis cell was sonicated (47 kHz) and the polarity of electrodes was alternated with intervals of 15 s. The optimized supplied electricity was found to be 6 F mol^{-1} based on **2** (entries 2–4). In this cross-coupling reaction the use of an excess amount of chlorodimethylsilane **1** relative to dichlorosilane **2** gave a better result (entries 1, 2), since a high concentration of **2** resulted in its oligomerization. The best yield of trisilane **3** was finally obtained when the solution of **2** in THF was added dropwise into the electrolyte during the reaction (entry 6). Under optimized reaction conditions the electroreductive cross-coupling reaction of **1** with diphenyldichlorogermane (**4**) was also carried out to form bis(dimethylsilyl)diphenylgermane (**5**) in good yield (Scheme 2).

2.2. Stepwise elongation of the Si–Si chain

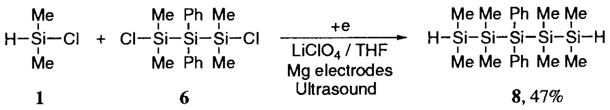
The Si–H bond was readily transformed to the Si–Cl bond by treatment with a catalytic amount of benzoylperoxide in carbon tetrachloride [7]. Using this method 1,3-dihydro-1,1,3,3-tetramethyl-2,2-diphenytrisilane (**3**) and bis(hydrodimethylsilyl)germane (**5**) were transformed into the corresponding chlorides **6** and **7**, respectively (Scheme 3). The further electroreduction of 1,3-dichloro-1,1,3,3-tetramethyl-2,2-diphenyltrisilane (**6**) with dimethylchlorosilane (**1**) gave the corresponding pentasilane **8** (Scheme 4), and these sequences were utilized for the synthesis of the odd-numbered oligosilanes. In the same manner, the even-numbered oligosilanes can be prepared. For instance, 1,4-dichloro-1,1,2,3,3,4,4-heptamethyl-2-phenyltetrasilane (**11**) can be prepared by the reaction between **1** and 1,2-dichloro-1,1,2-trimethyl-1-phenyldisilane (**9**) (Scheme 5). Accordingly, the electroreductive cross-



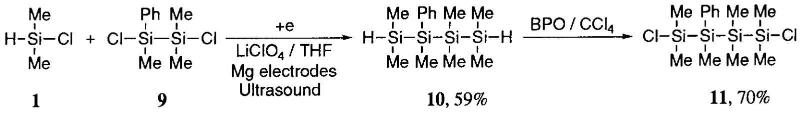
Scheme 2.



Scheme 3.



Scheme 4.



Scheme 5.

coupling reactions followed by the chlorination provided a powerful method for the stepwise elongation of the Si–Si bonds and synthesis of sequence-controlled oligosilanes.

2.3. *Electroreductive polymerization of 1,2-dichloro-1,1,2-trimethyl-1-phenyldisilane (**9**)*

The electroreductive polymerization of the dichlorooligosilanes is highly promising for the synthesis of sequence-ordered polysilanes. The electroreduction of dichlorodisilane **9** was found to give the corresponding polysilane **12** consisting of disilane units (Scheme 6). The electroreductive polymerization was carried out under a variety of reaction conditions, however, the yield of the resulting polymer was very low (Table 2) and low molecular weight oligomers ($M_n < 1000$) were formed. This is probably due to high reactivity of the disilene intermediate formed by the electroreduction of **9**. In fact, the addition of naphthalene, which could mask disilene [8], into the reaction system remarkably increased the yield of the polysilane **12** (entry 6).

2.4. *Electroreductive polymerization of dichlorooligosilanes*

Dichlorooligosilanes, such as dichlorotrisilane **6** were found to be good monomers for the electroreductive synthesis of the polysilanes having longer sequence units (Scheme 7). As summarized in Table 3, the temperature control is found to be very important in the electroreductive polymerization of **6**. The reaction at higher temperature, the backbiting reaction [9] of the propagating polymer proceeded forming cyclohexasilane as a by-product (entry 1). This side reaction was successfully suppressed when the reaction was carried out below 0°C, and polysilanes **13** having relatively high molecular weight were obtained (entries 3, 4). Under the optimized reaction conditions, the electroreduction of dichlorotetrasilane **11** gave the corresponding polysilane **14**, units of which were ordered in four sequences in satisfactory yield (Scheme 8). Moreover, the polymerization of bis(chlorodimethylsilyl)-diphenylgermane (**7**) gave the corresponding structure-controlled silane–germane copolymers **15** having the Si–Ge–Si sequences [10] (Scheme 9). The polymerizability of dichlorooligosilanes under electroreduction conditions seems to be mainly affected by the substituents on the chlorinated terminal silicon atom, and this fact provides possibilities for designing oligosilane sequences of internal silicon and germanium atoms.

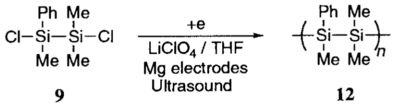
3. Experimental

IR spectra were obtained on a Hitachi 260-10 spectrometer. ^{1}H-NMR and ^{13}C-NMR spectra were measured on a Varian Gemini-200 (200 MHz) spectrometer, and the chemical shifts were referenced to $CDCl_3$ peaks. GC analyses were carried out on a Shimadzu GC-4C or GC-12A instrument. The gel permeation chromatography (GPC) system consisted of a Shimadzu LC-6A liquid chromatograph, a Shimadzu SP13-6AV UV–vis spectrophotometric detector, and a Shodex® GPC A-803 column. Molecular weight values are relative to the polystyrene standards [Shodex® STANDARD (SL-105) polystyrene]. The constant electrocurrent was supplied using a Takasago GPO 50-2 regulated DC power supply. The supplied electricity was counted by a Hokuto Denko Coulomb Amperehour Meter HF-201. The sonication of ultrasound (47 kHz) was performed using a Yarnato Branson 2200.

Chlorodimethylsilane (**1**), dichlorodiphenylsilane (**2**) and dichlorodiphenylgermane (**4**) are commercially available from Shin-Etsu Chemical Co. Ltd. and they were used after distillation. 1,2-Dichloro-1,1,2-trimethyl-2-phenyldisilane (**9**) was prepared using a reported method [11]. THF was distilled over Na–benzophenoneketyl under nitrogen atmosphere. Benzoyl peroxide (BPO) was recrystallized from benzene–ethanol. Magnesium ingot is commercially available from Rare Metallic Co. Ltd. and was cut into rods ($\Phi = 9$ mm, length $= 4$ cm) for electrodes. Mg electrodes were treated with concentrated HCl, and then washed with water and acetone.

3.1. *Electroreductive cross-coupling reaction of chlorodimethylsilane (**1**) with dichlorodiphenylsilane (**2**)*

The electroreduction was carried out in a 30-ml, three-necked flask equipped with an Mg cathode and



Scheme 6.

Table 2
Electroreductive polymerization of dichlorodisilane **9** [a]

Entry	Dichlorodisilane **9** (mol l^{-1})	Electricity (F mol^{-1})	Polysilane **12**		
			$\bar{M}_n$	$\bar{M}_w/\bar{M}_n$	Yield (%) [b]
1	0.11	4	2900	2.7	2.7
2	0.33	4	3600	2.3	2.4
3	0.67	4	2100	2.9	1.0
4	0.33	2	2800	2.7	3.9
5	0.33	6	3000	3.0	1.0
6 [c]	0.33	4	2500	1.81	3.0

[a] The electroreduction was carried out in a single-compartment cell using Mg electrodes under sonication (47 kHz), and the polarity of electrodes was alternated with an interval of 15 s.

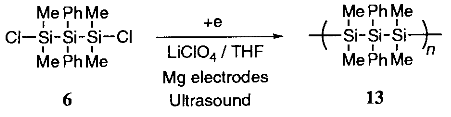
[b] Isolated.

[c] The electroreduction was carried out in the presence of naphthalene.

anode, and a three-way stopcock jointed to a balloon of nitrogen. Into this cell was placed 0.7 g of $LiClO_4$ and the content of the cell was dried at 50°C in vacuo for 3 h. Chlorotrimethylsilane (0.1 ml) and 15 ml, of dry THF were then added under a nitrogen atmosphere. After the solution was magnetically stirred for 3 h, the pre-electrolysis was carried out to remove traces of water and residual chlorotrimethylsilane from the electrolysis system. That is, 600 C of electricity was passed through the cell under the constant current condition (50 mA) and the polarity of the electrodes was alternated with an interval of 15 s using a comutator. During the electrolysis, the ultrasound (47 kHz) was sonicated while cooling with water. Chlorodimethylsilane (**1**, 5 mmol) and dichlorodiphenylsilane (**2**, 35 mmol) were then syringed into the cell in a stream of nitrogen, and the solution was further electrolyzed. The progress of the reaction was monitored by GLC or TLC. After the starting material was consumed (supplied electricity = ca. 6 F mol^{-1} based on **2**), the reaction mixture was poured into ice cold 1 N HCl (100 ml) and the aqueous solution was extracted with ether (50 ml × 3). The combined organic layer was washed twice with 50 ml of brine, dried over $MgSO_4$, and concentrated. The residue was purified by a silica gel column, eluting with hexane, and then distilled under reduced pressure (b.p. 136°C/37 mHg) to afford 1,1,3,3-tetramethyl-2,2-diphenyltrisilane (**3**). IR (neat) 3067, 2960, 2862, 2095, 1484, 1247, 838, 737 cm^{-1}; ^{1}H-NMR ($CDCl_3$) δ 0.30 (d, 12H, $J = 4.5$ Hz), 4.13 (sep, 2H, $J = 4.5$ Hz), 7.30–7.40 (m, 6H), 7.46–7.54 (m, 4H); MS m/z (relative intensity) 300 [4, M^+], 285 [3, $M^+ - Me$], 241 (31), 197 (41), 164 (85), 135 (82), 105 [100, $PhSi^+$], Anal. Calc. for $C_{16}H_{24}Si_3$: C, 63.93; H, 8.05. Found: C, 63.99; H, 7.98%.

3.2. Chlorination of 1,1,3,3-tetramethyl-2,2-diphenyltrisilane (3)

A solution of trisilane (**3**, 30 mmol) in CCl_4 (30 ml) and benzoyl peroxide (0.6 mmol) as an inductor were



Scheme 7.

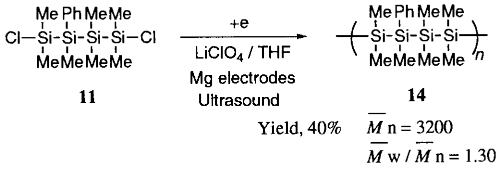
Table 3
Electroreductive polymerization of dichlorotrisilane **6** [a]

Entry	Reaction temperature	$\bar{M}_n$	$\bar{M}_w/\bar{M}_n$	Yield of **13** (%) [b]
1	18	3800	1.44	(42) [c]
2	0	4700	1.87	50
3	−10	5500	1.54	35
4	−15	4400	1.42	16

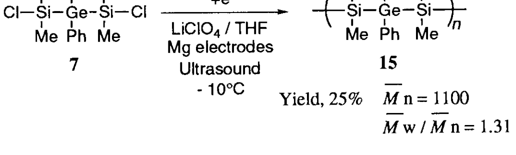
[a] The electroreduction was carried out by using Mg electrodes, and ultrasound (47 kHz) was applied during the reaction. The anode and cathode were alternated with an interval of 15 s. [dichlorotrisilane **6**] = 0.27 mol l^{-1}; [$LiClO_4$] = 0.35 mol l^{-1}; supplied electricity, 4 F mol^{-1}.

[b] Purified by reprecipitation from benzene–EtOH.

[c] Contaminated by 1,1,2,2,4,4,5,5-octamethyl-3,3,6,6-tetraphenylcyclohexasilane.



Scheme 8.



Scheme 9.

added into a round-bottomed flask equipped with reflux condenser jointed to a balloon of nitrogen, and refluxed for 2 h. Then, the resulting oil was concentrated and distilled under reduce pressure (b.p. 159°C/15 mHg) to give 1,3-dichloro-1,1,3,3-tetramethyl-2,2-diphenyltrisilane (**6**). IR (neat) 3070, 2954, 2893, 1483, 1250, 834, 737 cm^{-1}; ^1H-NMR ($CDCl_3$) δ 0.60 (s, 1211), 7.30–7.49 (m, 6H), 7.57–7.61 (m, 4H). HRMS *m*/*e* 368.03921 [Calc. for $C_{16}H_{22}Cl_{12}Si_3$, 368.04064].

*3.3. Electroreductive cross-coupling reaction of chlorodimethylsilane (**1**) with dichlorodiphenylgermane (**4**) and other dichlorooligosilanes (**6**, **9**)*

A solution of chlorosilane (**1**, 5 mmol) with dichlorodiphenylgermane (**4**, 35 mmol) or dichlorooligosilane (**6** or **9**) in dry THF (15 ml) was electrolyzed by using the same procedure as for the preparation of trisilane **3** to afford the corresponding dichlorooligosilanes (supplied electricity = ca. 6 F mol^{-1} based on **4**). The product was purified by a silica gel column, eluting with hexane.

Bis(dimethylsilyl)diphenylgermane (**5**): IR (neat) 3005, 2104, 1422, 1244, 1092, 878 cm^{-1}; ^1H-NMR ($CDCl_3$) δ 0.31 (d, 12H, $J = 4.3$ Hz), 4.26 (sep, 2H, $J = 4.3$ Hz), 7.28–7.33 (m, 6H), 7.40–7.45 (m, 4H); HRMS *m*/*e* 346.06196 [Calc. for $C_{16}H_{24}GeSi_2$, 346.06283].

1,1,2,2,4,4,5,5-Octamethyl-3,3-diphenylpentasilane (**8**): IR (neat) 3052, 2957, 2896, 2100, 1483, 1250, 838, 735 cm^{-1}; ^1H-NMR ($CDCl_3$) δ 0.01 (d, 12H, $J = 4.5$ Hz), 0.31 (s, 12H), 3.78 (sep, 2H, $J = 4.5$ Hz), 7.31–7.38 (m, 6H), 7.48–7.59 (m, 4H). Anal. Calc. for $C_{20}H_{36}Si_5$: C, 57.62; H, 830. Found: C, 57.88; H, 839%.

1,1,2,2,3,4,4-Hexamethyl-3-phenyltetrasilane (**10**): IR (neat) 3052, 2093, 1598, 1249, 838 cm^{-1}; ^1H-NMR (CDl_3) δ 0.02–0.32 (m, 21H), 3.72–3.86 (m, 1H), 3.88–4.04 (m, 1H), 7.26–7.58 (m, 5H).

*3.4. Chlorination of bis(dimethylsilyl)diphenylgermane (**5**) [10]*

Bis(dimethylsilyl)diphenylgermane (**5**) was chlorinated using the same procedure as for the chlorination of trisilane **3** to give bis(chlorodimethylsilyl)diphenylgermane (**7**). IR (neat) 3005, 2890, 735 cm^{-1}; ^1H-NMR ($CDCl_3$) δ 0.64 (s, 12H), 7.32–7.39 (m, 6H), 7.48–7.56 (m, 4H).

*3.5. Electroreductive polymerization of dichlorodisilane **9** and dichlorotrisilane **6***

Into the electrolysis cell (30-ml three-necked flask equipped with one pair of Mg electrodes and a three-way stopcock jointed to a balloon of nitrogen), was placed 1 g of $LiClO_4$. and the cell was dried at 50°C in vacuo for 3 h. Chlorotrimethylsilane (0.1 ml) and 15 ml of dry THF were then added under a nitrogen atmosphere. After pre-electrolysis was carried out using the same procedure as for trisilane, 3–5 mmol of the monomer (**9** or **6**) was syringed into the cell in a stream of nitrogen. The electroreduction was performed under constant current conditions (50 mA), and the polarity of the electrodes was alternated with an interval of 15 s using a comutator. During the electrolysis the ultrasound (47 kHz) was sonicated while cooling with water. The supplied electricity was counted by a comutator. After 4 F mol^{-1} of electricity was passed through the cell, the reaction was quenched by EtOH (10 ml). The mixture was then poured into ice-cold 1 N HCl (100 ml) and the aqueous solution was extracted with ether (50 ml × 3). The combined organic layer was washed twice with 50 ml of brine, dried over $MgSO_4$, and concentrated. The resulting crude polymer was dissolved in 4 ml of benzene and reprecipitated from EtOH (100 ml). The molecular weight of the polymer was determined by GPC with THF as eluent.

Poly[1,2,2-trimethyl-l-phenyldisilane] (**12**): IR (KBr) 3300, 1652, 1250, 710 cm^{-1}; ^1H-NMR ($CDCl_3$) δ 0.02–0.11 (m, 9H), 7.25–7.35 (m, 5H).

Poly[1,1,3,3-tetramethyl-2,2-diphenyltrisilane] (**13**): IR (KBr) 3308, 2981, 1428, 1250, 804, 702 cm^{-1}; ^1H-NMR ($CDCl_3$) δ 0.02–0.10 (m, 12H), 7.10–7.45 (m, 5H).

Poly[1,1,3,3,4,4-hexamethyl-2,2-diphenyltetrasilane] (**14**): IR (ICBr) 3156, 2958, 1382, 1299, 808 cm^{-1}; ^1H-NMR ($CDCl_3$) δ −0.30 to 0.20 (m, 18H), 7.00–7.40 (m, 5H).

Poly[bis(dimethylsilyl)diphenylgermane] (**15**): IR (KBr) 3068, 1431, 1260, 710, 1088 cm^{-1}; ^1H-NMR ($CDCl_3$) δ −0.54 to 0.47 (m, 12H), 6.29–7.52 (m, 5H).

Acknowledgements

The author (S.K.) thanks the Ministry of Education, Science and Culture Japan for Grant-in Aid for Scientific Research on Priority Areas (No. 11120257).

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