t, 1 H, HC=C(Si), J = 3.8 Hz), 7.29–7.90 (m, 4 H, phenyl ring protons); ^{13}C NMR (δ, in $CDCl_3$) −1.0 (Me_2Si), 1.4 (Me_2Si), 20.9, 22.8 (CH_2CH_2), 25.7 (Me_2C), 65.6, 65.7 (CH_2O and $CH(O)Me_2$), 112.3 (HC=C(Si)), 127.8, 128.1, 134.7, 136.2, 143.3, 145.2 (phenyl ring carbons), 160.3 (C(O)=C). Anal. Calcd for $C_{18}H_{30}O_2Si_2$: C, 64.61; H, 9.04. Found: C, 64.44; H, 8.80.

The retention time for GLC and the mass spectrum of dimethylphenylsilane were identical with those of an authentic sample.

Photolysis of 2 in the Presence of Isobutene. A mixture of 0.3669 g (1.33 mmol) of **2**, 1.18 g (21.1 mmol) of isobutene, and 0.0819 g (0.64 mmol) of tridecane in 20 mL of hexane was photolyzed for 45 min. The GLC analysis of the mixture showed the presence of dimethylphenylsilane (10% yield), **12** (7% yield), and the starting **2** (9% yield). Product **12** was isolated by preparative GLC: MS *m/e* 332 (M^+); IR 1635, 1428, 1372, 1248, 1153, 1108 cm^{-1}; ^{1}H NMR (δ, in C_6D_6) 0.02 (s, 3 H, MeSi), 0.14 (s, 3 H, MeSi), 0.41 (s, 3 H, MeSi), 0.54 (s, 3 H, MeSi), 0.89–2.07 (m, 5 H, CH_2CH_2CHSi), 1.60 (br s, 2 H, CH_2Si), 1.67 (br s, 3 H, MeC=C), 3.20–4.07 (m, 2 H, CH_2O), 3.48 (br d, 1 H, HC(O)Si, J = 2.9 Hz), 4.63 (br s, 1 H, H_aCH=C), 4.75 (br s, 1 H, H_bCH=C), 7.11–7.71 (m, 5 H, phenyl ring protons); ^{13}C NMR (δ, in C_6D_6) −3.6 (MeSi), −3.2 (MeSi), −0.3 (Me_2Si), 25.3 ($CHSiMe_2Ph$), 26.1, 26.4 (Me and CH_2Si), 27.1, 29.6 (CH_2CH_2), 71.0 (CH_2O), 77.4 (CH(Si)O), 109.0 (CH_2=C), 128.0, 129.0, 134.3, 140.0 (phenyl ring carbons), 143.4 (C(Me)=C). Anal. Calcd for $C_{19}H_{32}OSi_2$: C, 68.61; H, 9.70. Found: C, 68.56; H, 9.70.

The retention time for GLC and the mass spectrum of dimethylphenylsilane were identical with those of an authentic sample.

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Electrochemistry of Organosilicon Compounds. 1. Synthesis of Di- and Trisilanes with Use of Mercury and Silver Electrode Systems

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Electrochemical formation of an Si–Si bond has been examined. Electrolysis of monochlorosilanes in 1,2-dimethoxyethane using a platinum cathode and a mercury anode gave disilanes in high yield, while the electrolysis of a mixture of two different monochlorosilanes produced unsymmetrical disilanes as main products. Similar treatment of a mixture of monochlorosilanes and dichlorosilanes gave trisilanes. The use of silver as the anode also gave the di- and trisilanes in high yields.

Introduction

In recent years, the rapid development of material science has led to extensive investigation of polymeric organosilicon compounds having silicon–silicon bonds in the polymer backbone. In this field, the silicon–silicon bond formation is of considerable importance for building up these molecules. However, the practical method used for the formation of the silicon–silicon bonds is limited to the alkali-metal condensation of chlorosilanes.[1–5]

A few papers concerning the silicon–silicon bond formation that involves no alkali-metal condensation have been published to date.[6–9] Hengge and his co-workers have reported that the electrochemical reduction of chlorosilanes using a Pt cathode and a Hg anode in dimethoxyethane led to the formation of coupling products.[6] Corriu et al. have demonstrated that chlorotriphenylsilane could be electrochemically coupled, but they found that large quantities of disiloxanes were formed even when the reaction was carried out under controlled-potential conditions.[7] More recently, Boudjouk has summarized the electroreductive coupling including dimerization of functionally substituted chlorosilanes,[8] but no detailed procedure is described. In this paper, we report electrochemical high-yield syntheses of disilanes and trisilanes from chlorosilanes.

Results and Discussion

The synthesis of polysilane oligomers by electrochemical reduction using mercury as a "sacrificial" anode was originally reported by Hengge and his co-workers.[6] First, we have followed this method for the Si–Si bond formation using chlorodimethylphenylsilane in an H-shaped divided cell having a sintered glass diaphragm, as reported by Hengge.[6] However, as the reaction progressed, mercury

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Table I. Formation of Polysilanes by Exhaustive Reduction of Chlorosilanes under Controlled-Current Conditions

chlorosilanes A	chlorosilanes B	ratio B/A	anode	products isolated/% yield[a] (current efficiency[c])
$Me_2PhSiCl$			Hg	**1**/84 (37)
$MePh_2SiCl$			Hg	**2**/89 (57)
$Me_2PhSiCl$	Me_3SiCl	2.1	Hg	**3**/72 (22), **1**/8 (1)
$MePh_2SiCl$	Me_3SiCl	2.2	Hg	**4**/94 (30), **2**/4 (1)
$MePh_2SiCl$	Me_2Bu^tSiCl	1.0	Hg	**5**/78 (30), **2**/17 (3)
Me_2SiCl_2	$MePh_2SiCl$	3.6	Hg	**6**/48 (6), **2**/54[b] (7)
$MePh_2SiCl$			Al	**2**/47 (40)
$MePh_2SiCl$			Ag	**2**/85 (62)
$MePh_2SiCl$	Me_3SiCl	2.5	Ag	**4**/78 (65)
$MePhSiCl_2$	Me_3SiCl	4.4	Ag	**7**/45 (33)

[a] Yield based on A unless otherwise noted. [b] Yield based on B. [c] Current efficiency was calculated by [mol of product × number of electron concerned/quantity of electricity supplied (F)] × 100.

salts deposited in large amounts onto the surface of the sintered glass diaphragm, leading to a lowering of the conductivity. As a result, the low conductivity of the electrolytic system prevented completion of the reaction and the coupling product, 1,2-diphenyltetramethyldisilane (**1**) was produced only in low yield. We found, however, that the use of an undivided cell and careful drying of the electrolytic system, including the reagents used, result in a high yield of the dimer **1**. The reaction mixture was analyzed by gas-phase chromatography (GPC) with the use of *n*-tetradecane as an internal standard. The reaction proceeds quite cleanly, and the current efficiency in the formation of the disilane **1** reaches 96% when 50% of the starting material is consumed. The formation of diphenyltetramethyldisiloxane was observed only in trace amounts during the electrolysis.

Therefore, we used the undivided cell equipped with a mercury pool as the anode (7 cm^2) and a platinum plate as the cathode (6 cm^2) in a manner described in the Experimental Section. Thus, electrolysis of chlorodimethylphenylsilane (10.53 mmol) was carried out in dimethoxyethane under controlled current (100 mA). At the point of passing 2.27 F/mol of the electricity, GPC analysis of the mixture showed that all of the chlorosilane had been consumed.[10] Mercury(I) chloride was filtered off and the filtrate was concentrated under reduced pressure. 1,2-Diphenyltetramethyldisilane (**1**) was isolated by medium-pressure liquid chromatography (MPLC) in 84% yield on the basis of the chlorosilane used. The amount of disiloxane was found to be less than 2%. Similarly, electrolysis of chloromethyldiphenylsilane gave 1,2-dimethyltetraphenyldisilane (**2**) in 89% yield. Results are summarized in Table I.

$R^1R^2MeSiSiMeR^3R^4$
1, $R^1 = R^3 = Me$, $R^2 = R^4 = Ph$
2, $R^1 = R^2 = R^3 = R^4 = Ph$
3, $R^1 = Ph$, $R^2 = R^3 = R^4 = Me$
4, $R^1 = R^2 = Ph$, $R^3 = R^4 = Me$
5, $R^1 = R^2 = Ph$, $R^3 = Me$, $R^4 = Bu^t$

$R^2R^3MeSiSiR^1MeSiMeR^2R^3$
6, $R^1 = Me$, $R^2 = R^3 = Ph$
7, $R^1 = Ph$, $R^2 = R^3 = Me$

Unsymmetrical disilanes can also be prepared by this method. Thus, the cross-coupling reaction of chlorodimethylphenylsilane with 2.1 molar equiv of chlorotrimethylsilane under the same conditions gave pentamethylphenyldisilane (**3**) in 72% yield, in addition to 8% of the homo-coupling product **1**. Similarly, the reaction of chloromethyldiphenylsilane with 2.2 molar equiv of chlorotrimethylsilane afforded 1,1-diphenyltetramethyldisilane (**4**) and compound **2** in 94% and 4% yields, respectively. The cross-coupling reaction proceeded selectively even when an equimolar mixture of two different chlorosilanes was used. Thus, electrolysis of an equimolar mixture of chloromethyldiphenylsilane and *tert*-butylchlorodimethylsilane gave 2-*tert*-butyl-1,1-diphenyltrimethyldisilane (**5**) and the disilane **2** in 78% and 17% yields, respectively.

The present method is not restricted to the synthesis of disilanes but can be used for the preparation of trisilanes. Electrolysis of a mixture of dichlorodimethylsilane in the presence of 3.6 molar equiv of chloromethyldiphenylsilane under the same conditions afforded compound **2** and 1,1,3,3-tetraphenyltetramethyltrisilane (**6**) in 54% and 48% yields, respectively.

In these reactions, mercury serves as the "sacrificial" anode and is oxidized to monovalent Hg during the electrolysis. Thus, the stoichiometric amount of mercury(I) chloride is produced after the electrolysis. Although the mercury(I) salt is insoluble and can readily be removed by filtration, it is desirable to carry out these reactions using a less toxic material. We therefore attempted to find an effective anode material other than mercury.

When the reductive coupling of chloromethyldiphenylsilane was attempted by using platinum as the anode material instead of mercury, product **2** was produced in low yield. It seems likely that undesirable oxidation of either the starting material or the product occurs on the anode surface or, more possibly, chlorine produced by anodic oxidation of chloride ions decomposes the disilane. The use of a suitable anodic partner that interrupts oxidation of chloride ion but does not interfere with the cathodic process is thought to be essential to obtain high yields of the disilane. The use of an alkaline-earth metal such as magnesium as the anode material seemed of interest, since they are oxidized at a much less positive potential than not only chloride ion but also mercury. When Mg was used as the anode, magnesium chloride formed in the solution was reduced on the cathode surface to form a thick insulating film, making the cathode passive.[11] On the other hand, when aluminum was used as the anode, the cathode did not become passive, but the disilane **1** was obtained only in 47% yield.[9]

We have found that silver can be used as an excellent anode material in place of mercury. Thus, the electrolysis of chloromethyldiphenylsilane in the undivided cell fitted with a silver wire as the anode and a platinum plate as the cathode afforded disilane **2** in 85% yield. During the electrolysis, the silver surface used as the anode was covered with an insoluble silver chloride layer, but neither the anode nor the cathode became passive. When the reduction of the same substrate, chloromethyldiphenylsilane, was carried out in the presence of 2.5 molar equiv of chlorotrimethylsilane, the cross-coupling product, disilane **4**, was obtained in 78% yield. The homo-coupling product **2** was produced in negligible amount. Similarly, the reaction of dichloromethylphenylsilane with 4.4 molar equiv of chlorotrimethylsilane gave 2-phenylheptamethyltrisilane (**7**) in 45% yield. The formation of 2,3-diphenyloctamethyltetrasilane was observed only in trace amounts.

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(10) In synthetic runs, high amounts of electricity were supplied for the completion of the reaction, since remaining chlorosilane causes the formation of disiloxane during workup.

(11) Recently, Shono and his co-workers have reported that disilanes are obtained from chlorosilanes by using magnesium for both the anode and the cathode and by alternating the direction of the current every 15 s: Shono, T.; Kashimura, S.; Nishida, R. 59th National Meeting of the Chemical Society of Japan, Yokohama, April 1990; Abstract No. 3D710.

All of the above results were quite reproducible and the formation of siloxanes was found to be less than a few percent.[12]

Experimental Section

General Data. The electrolysis of chlorosilanes was carried out in a 25-mL undivided cell equipped with a platinum plate (6 cm^2) as the cathode and a mercury pool (7 cm^2) as the anode. In experiments using siliver or aluminum as the anode, a silver wire (10.5 cm^2) of 1-mm diameter or an aluminum plate (15 cm^2) was used together with the platinum cathode. In a 25-mL cell was placed 1.25 g of tetrabutylammonium perchlorate, and the cell was dried at 50 °C in vacuo for 3 h to remove traces of water from the inside. Chlorosilane and 20 mL of 1,2-dimethoxyethane were then added under a dry nitrogen atmosphere. Electrolysis was carried out in a manner of controlled current (100 mA for Hg and 50 mA for both Ag and Al). The progress of the reaction was monitored by GPC and the electrolysis was continued until all of the starting material was consumed. The resulting solution was concentrated under reduced pressure. Products were isolated by MPLC (silica gel 40–63 μm), eluting with hexane or a mixture of hexane–dichloromethane (9:1).

The structures of the polysilanes obtained were characterized by spectroscopic methods as well as by elemental analysis. All the spectral data obtained for the polysilanes were identical with those reported in the literatures. In the reactions in which two different chlorosilanes were used, the yields were calculated on the basis of the chlorosilane, which was used in the smaller molar amount.

Materials. Chloromethyldiphenylsilane, chlorodimethylphenylsilane, chlorotrimethylsilane, *tert*-butylchlorodimethylsilane, dichlorodimethylsilane, and dichloromethylphenylsilane were supplied by Shin-Etsu Chemical Co. Ltd. and were distilled before use. 1,2-Dimethoxyethane was refluxed over $LiAlH_4$ or Na–benzophenone under a nitrogen atmosphere and distilled into a vessel containing Molecular Sieves-4A that had been preliminarily dehydrated in vacuo at 350 °C for 4 h. This solvent was kept overnight before use. Tetrabutylammonium perchlorate was synthesized by the reaction of tributylamine and butyl bromide and subsequent treatment with perchloric acid. The product was recrystallized from acetone–water (5 times) and dried in vacuo at 50 °C overnight.

Reduction of Chlorosilanes. (a) Chlorodimethylphenylsilane. Chlorodimethylphenylsilane (10.53 mmol) was electrolyzed by using a Hg anode (23.9 mF) to give 1,2-diphenyltetramethyldisilane[13] (1) (1.190 g, 4.40 mmol, 84%).

For 1: ^{1}H NMR (δ in $CDCl_3$) 0.35 (s, 12 H, Me), and 7.2–7.65 ppm (m, 10 H, Ph); ^{13}C NMR (δ in $CDCl_3$) –3.82 (Me), 127.71, 128.41, 133.88, and 138.98 ppm (Ph); MS *m/e* 270 (M^+) and 135 ($PhMe_2Si^+$).

(b) Chloromethyldiphenylsilane. Chloromethyldiphenylsilane (8.87 mmol) was electrolyzed by using a Hg anode (13.9 mF) to give 1,2-dimethyltetraphenyldisilane[13] (2) (1.566 g, 3.97 mmol, 89%). The electrolysis (9.78 mF) of the same chlorosilane (7.13 mmol) using a Ag anode afforded the disilane **2** (1.20 g, 3.04 mmol, 85%). The electrolysis (6.18 mF) of the chlorosilane (5.29 mmol) using an Al anode afforded the disilane **2** (0.492 g, 1.25 mmol, 47%).

For 2: ^{1}H NMR (δ in $CDCl_3$) 0.69 (s, 6 H, Me), and 7.15–7.65 ppm (m, 20 H, Ph); ^{13}C NMR (δ in $CDCl_3$) –4.04 (Me), 127.82, 128.90, 135.18, and 136.59 ppm (Ph); MS *m/e* 394 (M^+) and 197 (Ph_2MeSi^+).

(c) Chlorodimethylphenylsilane and Chlorotrimethylsilane. Chlorodimethylphenylsilane (3.73 mmol) and chlorotrimethylsilane (7.93 mmol) were electrolyzed by using a Hg anode (24.9 mF) to give phenylpentamethyldisilane[14] (3) (0.562 g, 2.70 mmol, 72%) and disilane 1 (0.038 g, 0.14 mmol, 8%).

For 3: ^{1}H NMR (δ in $CDCl_3$) 0.07 (s, 9 H, Me), 0.32 (s, 6 H, Me), and 7.20–7.55 ppm (m, 5 H, Ph); ^{13}C NMR (δ in $CDCl_3$) –3.93 (Me_2), –2.20 (Me_3), 127.76, 128.30, 133.77, and 139.63 ppm (Ph); MS *m/e* 208 (M^+), 193 (M^+ – Me), 135 ($PhMe_2Si^+$), and 73 (Me_3Si^+).

(d) Chloromethyldiphenylsilane and Chlorotrimethylsilane. Chloromethyldiphenylsilane (4.58 mmol) and chlorotrimethylsilane (10.3 mmol) were electrolyzed by using a Hg anode (29.1 mF) to give 1,1-diphenyltetramethyldisilane[14] (4) (1.166 g, 4.31 mmol, 94%) and disilane **2** (0.035 g, 0.09 mmol, 4%). The electrolysis (9.84 mF) of these chlorosilanes (4.08 and 10.3 mmol) using a Ag anode afforded 4 (0.866 g, 3.20 mmol, 78%).

For 4: ^{1}H NMR (δ in $CDCl_3$) 0.19 (s, 9 H, Me), 0.62 (s, 3 H, Me), and 7.20–7.65 ppm (m, 10 H, Ph); ^{13}C NMR (δ in $CDCl_3$) –4.85 (Me), –1.66 (Me_3), 127.82, 128.68, 134.80, and 137.46 ppm (Ph); MS *m/e* 270 (M^+), 255 (M^+ – Me), 197 (Ph_2MeSi^+), 135, 105, and 73 (Me_3Si^+).

(e) Chloromethyldiphenylsilane and *tert*-Butylchlorodimethylsilane. Chloromethyldiphenylsilane (5.25 mmol) and *tert*-butylchlorodimethylsilane (5.18 mmol) were electrolyzed by using a Hg anode (27.2 mF) to give 2-*tert*-butyl-1,1-diphenyltrimethyldisilane (5) (1.282 g, 4.10 mmol, 78%) and disilane **2** (0.176 g, 0.446 mmol, 17%).

For 5: ^{1}H NMR (δ in $CDCl_3$) 0.16 (s, 6 H, Me), 0.71 (s, 3 H, Me), 0.87 (s, 9 H, Bu^t), 7.21–7.42 (m, 6 H, $Ph_{o,p}$), and 7.42–7.68 ppm (m, 4 H, Ph_m); ^{13}C NMR (δ in $CDCl_3$) –5.07 (Me_2), –3.17 (Me), 18.22 (*C*–Me_3), 27.76 (Me_3), 127.76, 128.63, 134.91, and 137.95 ppm (Ph); MS *m/e* 312 (M^+), 255 (M^+ – Bu^t), 197 (Ph_2MeSi^+), 135, 105, and 73. Anal. Calcd for $C_{19}H_{28}Si_2$: C, 73.00; H, 9.03. Found: C, 73.00; H, 9.02.

(f) Dichlorodimethylsilane and Chloromethyldiphenylsilane. Dichlorodimethylsilane (2.09 mmol) and chloromethyldiphenylsilane (7.52 mmol) were electrolyzed by using a Hg anode (62.2 mF) to give 1,1,3,3-tetraphenyltetramethyltrisilane[15] (6) (0.454 g, 1.00 mmol, 48%) and disilane **2** (0.779 g, 2.02 mmol, 54% based on the monochlorosilane).

For 6: ^{1}H NMR (δ in $CDCl_3$) 0.28 (s, 6 H, Me), 0.46, (s, 6 H, Me), and 7.18–7.50 ppm (m, 20 H, Ph); ^{13}C NMR (δ in $CDCl_3$) –4.96 (Me), –4.20 (Me), 127.76, 128.74, 134.91, and 137.13 ppm (Ph); MS *m/e* 452 (M^+), 255 (M^+ – $MePh_2Si$), 240 (M^+ – Me), 197 (Ph_2MeSi^+), 178, 135, and 105.

(g) Dichloromethylphenylsilane and Chlorotrimethylsilane. Dichloromethylphenylsilane (1.72 mmol) and chlorotrimethylsilane (7.64 mmol) were electrolyzed by using a Ag anode (9.33 mF) to give 2-phenylheptamethyltrisilane[16] (7) (0.207 g, 0.776 mmol, 45%).

For 7: ^{1}H NMR (δ in $CDCl_3$) 0.13 (s, 18 H, Me), 0.40, (s, 3 H, Me), and 7.18–7.50 ppm (m, 5 H, Ph); ^{13}C NMR (δ in $CDCl_3$) –8.97 (Me), –1.01 (Me_6), 127.71, 134.48, and 137.62 ppm (Ph); MS *m/e* 266 (M^+), 251 (M^+ – Me), 193 (M^+ – Me_3Si), 135, and 73 (Me_3Si^+).

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