

Friedel–Crafts alkylation of benzene with (polychloromethyl)silanes

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Abstract

(Polychloromethyl)silanes ($\text{Cl}_{3-m}\text{Me}_m\text{SiCH}_3-n\text{Cl}_n$; $m = 0-3$; $n = 2, 3$) reacted with excess benzene in the presence of aluminum chloride to give (polyphenylmethyl)silanes. Such reactions occurred at the temperatures ranging from room temperature ($m = 2, 3$; $n = 2$) to 80°C ($m = 0, 1$; $n = 2, 3$), indicating that the reactivity increases with increasing the number (m) of electron-donating methyl-group(s) at the silicon. In particular, (dichloromethyl)silanes with two or three methyl groups at the silicon ($m = 2$ or 3 ; $n = 2$) underwent the alkylation and the decomposition of their products at room temperature. The reaction with (dichloromethyl)trimethylsilane occurred immediately at room temperature to give no (diphenylmethyl)trimethylsilane, but diphenylmethane and trimethylchlorosilane via the decomposition of alkylation product. (Trichloromethyl)silanes ($m = 0, 1$; $n = 3$) reacted with excess benzene to give (triphenylmethyl)silanes as major products and the unusual (diphenylmethyl)silanes as minor. It was found that unusual (diphenylmethyl)silanes were formed by the decomposition of (triphenylmethyl)silanes under the reaction condition. In the alkylation to benzene, the reactivity of (polychloromethyl)silanes ($\text{Cl}_{3-m}\text{Me}_m\text{SiCH}_3-n\text{Cl}_n$; $m = 0-3$; $n = 2, 3$) decreases in the following order: $m = 3 > 2 > 1 > 0$; $n = 3 > 2$. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

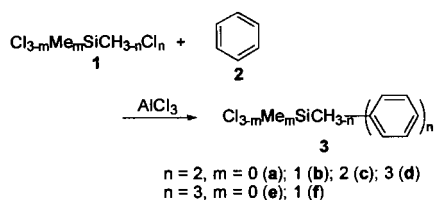
Friedel–Crafts alkylations of aromatic compounds using a wide variety of alkylating reagents such as alkyl halides, alkene and, etc. for a long time have been used both in the laboratory and in the petroleum industry [1,2]. While the alkylations with organosilicon compounds [3] such as alkenylchlorosilanes [4] and (chloroalkyl)chlorosilanes [5] have been received little attention due to the difficulty of their syntheses and separation [6], even though organosilicon compounds with Si–Cl bond(s) as functionality are well known to be potential starting materials for binders, rubber, resin, oil, and etc. in industrial field.

In 1993, allyldichlorosilane was successfully prepared by the direct reaction of elemental silicon with allyl

chloride [7]. In the viewpoint of its application, we started the Friedel–Crafts alkylation of benzene derivatives with allyldichlorosilanes [8] and then have studied the alkylation with other organosilanes such as allylchlorosilanes [9], methylvinylidichlorosilane [10], and (ω -chloroalkyl)chlorosilanes [11] in the presence of aluminum chloride catalyst. So far, such reactions were focused on the alkylation with simple monofunctionalized organosilanes. In the extension of this alkylation, polyfunctionalized organosilanes, (polychloromethyl)silanes, instead of monofunctionalized organosilanes were employed for the alkylation in the presence of aluminum chloride. Friedel–Crafts alkylation of (Polychloromethyl)silanes [$\text{Cl}_{3-m}\text{Me}_m\text{SiCH}_3-n\text{Cl}_n$; $m = 0-3$; $n = 2, 3$] to benzene (**2**), known to be the simplest aromatic compound, was carried out in the presence of aluminum chloride catalyst. In this paper, we wish to report the results observed from the Friedel–Crafts alkylation of (polychloromethyl)silanes to excess **2** in details.

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Scheme 1.

Table 1
Alkylation conditions^a of **2** with (dichloromethyl)silanes (**1a–d**) and product yields

1 ^b	Reaction conditions		Products (%) ^c
	Temp. (°C)	Time (h)	3
1a (89)	80	3.0	3a (7) ^d
1b (–)	80	0.5	3b (75, 87%)
1c (85)	r.t.	2.0	3c (10)
1c (–)	80	0.5	3c (20), others ^{d,f}
1d (6)	r.t.	0.5	3d (–), others ^{d,g}

^a 10:1 Reaction of **2** with (dichloromethyl)silanes **1a–d** was carried out in the presence of 20 mol% AlCl_3 based on **1** used.

^b Unreacted **1** is in parenthesis.

^c Isolated yield if not described.

^d GLC yield.

^e Reaction was carried out in the presence of 10 mol% AlCl_3 .

^f A 2:1 mixture of dimethyldichlorosilane to diphenylmethane was observed as predominant products.

^g A 3:1 mixture of trimethylchlorosilane to diphenylmethane as predominant products.

2. Results and discussion

The reaction of (polychloromethyl)silanes **1** [$\text{Cl}_{3-m}\text{Me}_m\text{SiCH}_2\text{-}_n\text{Cl}_n$; $m = 0–3$; $n = 2, 3$] with the simplest **2** as a representative among aromatic compounds was carried out in the presence of aluminum chloride. (Polyphenylmethyl)chlorosilanes which contain Si–Cl bond(s) as functionality were obtained from the reaction with (polychloromethyl)silanes containing one or more chlorine-substituent(s) at the silicon (Scheme 1).

2.1. Alkylation with (dichloromethyl)silanes

The alkylation of **2** with simple (dichloromethyl)silanes (**1a–d**) as representative among (polychloromethyl)silanes in the presence of aluminum chloride catalyst was carried out to study the effect on the substituents at the silicon atom. In this reaction, compound **2** was used with ten times excess respect to **1** to protect the second alkylation to the phenyl-ring of alkylation products. Under this reaction condition, such alkylation products were not obtained in appreciated amounts.

The results obtained from the alkylation reaction of (dichloromethyl)silanes **1a–d** to excess **2** are summarized in Table 1.

As shown in Table 1, the alkylation of **2** with (dichloromethyl)silanes **1a–d** occurred at the temperatures ranging from room temperature to 80°C. In the alkylation to **2**, 11% of (dichloromethyl)trichlorosilane (**1a**) substituted with all chloro-groups at the silicon was consumed at 80°C for 3 h to give product **3a** in 7% yield. (Dichloromethyl)methyldichlorosilane (**1b**) substituted with two chloro- and methyl-groups at the silicon reacted with **2** at 80°C for 0.5 h to give product **3b** in 75 and 87% yields when 20 and 10 mol% of aluminum chloride were used as catalyst, respectively. While the reaction of **2** with (dichloromethyl)silanes, substituted with two or more methyl-groups at the silicon, such as (dichloromethyl)dimethylchlorosilane (**1c**) and (dichloromethyl)trimethylsilane (**1d**) occurred at room temperature, but the reaction with **1c** was carried out at 80°C because the consumption of **1c** was too slow. In this reaction, alkylation product **3c** was obtained in 20% yield as minor along with decomposed products, dimethyldichlorosilane and diphenylmethane. In particular, the reaction with **1d** occurred immediately at room temperature to give trimethylchlorosilane and diphenylmethane without an expected alkylation product, (diphenylmethyl)trimethylsilane (**3d**). Such decomposition products, trimethylchlorosilane and diphenylmethane, can be formed from the decomposition of **3d** by HCl in the presence of aluminum chloride as observed in other similar system [11]. These results indicate that electron-donating methyl-group(s) at the silicon of **1** facilitate the alkylation to **2** and also the carbon–silicon bond cleavage reaction of alkylation products [11]. The effect of the substituents at the silicon of (dichloromethyl)silanes on the alkylation to **2** can be rationalized in the term of the stability of carbocation intermediate [5e,12], generated by the complexation reaction of (dichloromethyl)silanes with aluminum chloride, attacking electrophilically to **2** [11]. These results showed that the reactivity of the alkylation to **2** increases as the number of chlorine-substituent(s) at the silicon of (dichloromethyl)silanes decreases from **1a** to **1c**.

2.2. Alkylation with (trichloromethyl)silanes

Based on the fact that (dichloromethyl)silanes substituted with two or more chloro-groups at the silicon give the stable alkylated products above, (trichloromethyl)chlorosilanes such as (trichloromethyl)trichlorosilane (**1e**) and (trichloromethyl)methyldichlorosilane (**1f**) as representatives among (trichloromethyl)chlorosilanes reacted with excess **2** in the presence of 20 mol% aluminum chloride at 80°C. These results are summarized in Table 2.

As shown in Table 2, on the basis of consumption of **1**, the reactivity of **1f** was slightly higher than that of **1e**

in the aluminum chloride-catalyzed alkylation and the yield of **3f** was also higher than **3e**. These results indicate that the electron donating methyl-substituent at the silicon of **1f** facilitates the alkylation than the electron withdrawing chlorine-substituent at that of **1e**, as observed in the reaction with (dichloromethyl)silanes **1a** and **1b**. In this reaction, triphenylated products of (triphenylmethyl)silanes **3e** and **3f** were obtained as the major products along with the unexpected diphenylated products of (diphenylmethyl)silane **3a** and **3b** as minor from the reaction with **1e** and **1f**, respectively. Especially, in the reaction with **1f**, 72 and 18% of products **3f** and **3b** obtained with the 93% consumption of **1f** in a 0.5 h reaction were changed to 68 and 25% of those in an 1 h reaction, respectively. It seems likely that (triphenylmethyl)silanes **3e** and **3f** are decomposed to (diphenylmethyl)silanes **3a** and **3b** in the reaction condition.

In order to test whether the (triphenylmethyl)chlorosilanes in the presence of aluminum chloride is decomposed to (diphenylmethyl)chlorosilanes in detail, compound **3f** was stirred under the same alkylation condition except for the starting material **1f**. In this reaction, compound **3f** was decomposed to give **3b**. The results obtained from the decomposition reaction of **3f** to **3b** in excess benzene in the presence of aluminum chloride are summarized in Table 3.

As shown in Table 3, the decomposition of **3f** did not occur at room temperature, but decomposed at the reflux temperature of benzene. In 1 and 2 h reactions,

Table 2
Alkylation conditions of benzene (**2**) with (trichloromethyl)silanes (**1e,f**) and product yields

1	Reaction conditions ^a		Products (%)
	Temp. (°C)	Time (h)	3
1e (7)	80	1.0	3e (60), 3a (2)
1e (–)	80	2.0	3e (64), 3a (3)
1f (7)	80	0.5	3f (72), 3b (18)
1f (–)	80	1.0	3f (68), 3b (25)

^a 15:1 Reaction of **2** with (trichloromethyl)silanes **1** was carried out in the presence of 20 mol% AlCl₃ based on **1** used.

Table 3
Decomposition of **3f** to **3b** in the presence of AlCl₃

Reaction conditions ^a		Compounds (%)	
Temp. (°C)	Time (h)	3f	3b
r.t.	2	100	–
80	1	85	10
80	2	72	20

^a The reaction was carried out in the presence of 20 mol% aluminum chloride catalyst.

15 and 28% of **3f** were consumed to give 10 and 20% of **3b**, respectively. The results showed that the decomposition occurs under the alkylation reaction condition of **2** with (trichloromethyl)silanes as observed in the decomposition reaction of tetraphenylmethane to triphenylmethane [13].

In conclusion, the reactivity of (chlorinated methyl)silanes in the aluminum chloride-catalyzed Friedel–Crafts alkylation to excess **2** increases as the number of chloro-groups on the carbon at the silicon atom increases from (chloromethyl)trichlorosilane [11] to **1a**, to **1e** in the case of (chlorinated methyl)trichlorosilanes. It seems likely that the reactivity order of this alkylation is consistent with a stabilization of carbocation intermediate (Cl–C⁺...–AlCl₄) by an electromeric electron-donating effect of the chlorine atom directly bonded to the carbon with cation, forming C–Cl⁺ intermediate as another resonance structure [5e]. The reaction was also affected on the electronic nature of the substituents at the silicon atom of (polychloromethyl)silanes. In the alkylation of (dichloromethyl)silanes **1a–d**, the reactivity increases in the following order: **1a** < **1b** < **1c** < **1d**, indicating that the alkylation is facilitated with increasing the number of electron donating methyl-substituent(s) at the silicon of **1**. While the decomposition of alkylation products was observed from the reaction with (dichloromethyl)silanes, substituted with two or more methyl-groups at the silicon, **1c** and **1d**: in particular, the alkylation with **1d** only gave the decomposed products, diphenylmethane and trimethylchlorosilane without expected alkylation product **2d**. The reaction of (trichloromethyl)silane **1e** and **1f** with excess **2** gave (triphenylmethyl)silanes **3e** and **3f** as major products and the unusual (diphenylmethyl)silanes **3a** and **3b** as minor, respectively. It was found that (diphenylmethyl)silanes were formed by the decomposition reaction of (triphenylmethyl)silanes under the alkylation reaction condition. In the alkylation to **2**, the reactivity of (polychloromethyl)silanes [Cl_{3–m}Me_mSiCH_{3–n}Cl_n; m = 0–3; n = 2, 3] decreases in the following order: m = 3 > 2 > 1 > 0; n = 3 > 2.

3. Experimental

All reactions and manipulations were carried out under prepurified dinitrogen using Schlenk techniques. Glassware was flame-dried before use. Dried solvents were employed in all reactions. Aluminum chloride and benzene were purchased from Aldrich Chemical Co. and all (dichloromethyl)silanes **1a–d** and (trichloromethyl)chlorosilane from Gelest Inc. and used without purification except for benzene. (Trichloromethyl)methyldichlorosilane was prepared by the known method [6c]. Benzene was dried by the distillation from

sodium benzophenone ketyl prior to use. The reaction products were analyzed by GLC using a packed column (10% SE-30 or SE-54 on 80–100 mesh chromosorb W/AW, 1/8 in. \times 1.5 m) or a capillary column (SE-30, 30 m) with a Varian 3300 gas chromatograph, thermal conductivity detector, and Hitachi D-2500 integrator. The progresses of the reactions were monitored by GLC. Product yields were determined chromatographically with *n*-dodecane as an internal standard, if not described in details. Samples for characterization were purified by a preparative GLC using a Donam system series DS 6200 gas chromatograph with a thermal conductivity detector and a 4 m by 1/8 in. stainless steel column packed with 20% OV-101 on 80–100 mesh chromosorb P/AW. NMR spectra were recorded on a Varian Gem 300 (FT, 300 MHz, ^1H ; 75 MHz, ^{13}C) or a Bruker Avance 300 spectrometers in CDCl_3 solvent. Mass spectra were obtained using a Hewlett Packard 6890 GC/MS. Elemental analyses for new compounds **3e** and **3f** were performed by the Chemical Analysis Center at the Korea Institute of Science and Technology.

3.1. Reaction of **2** with **1a**

To a 50 ml flask was charged **1a** (3.28 g, 15.0 mmol), **2** (11.7 g, 150.0 mmol), AlCl_3 (0.4 g, 3.0 mmol), and dodecane (0.5 g) under nitrogen atmosphere at room temperature (r.t.). The reaction mixture was stirred at 80°C for 1 h. The product **3a** (7%) was observed with the 11% consumption of **1a** by a GLC.

Spectral data for **3a**: $^1\text{H-NMR}$ (300 MHz) δ 4.28 (s, 1H, SiCH), 7.31–7.48 (m, 10H, phenyl-*H*); $^{13}\text{C-NMR}$ (75 MHz) δ 50.39 (SiCH), 127.12, 128.86, 129.41, 136.60 (phenyl-carbons). Mass spectrum: m/z (relative intensity) 304 (2), 302 (6), 300 (6) (M^+), 168 (14), 167 (100, Ph_2HC^+), 166 (11), 165 (34), 152 (14). For other physical properties, refer to the previous report [5b].

3.2. Reaction of **2** with **1b**

Using the procedure described in the reaction of **2** with **1a**, the reactions of **2** (12.0 g, 153.6 mmol), with **1b** (3.0g, 15.2 mmol) in the presence of aluminum chloride (0.4 g, 3.0 mmol) were carried out at 80°C for 0.5 h. Then, NaCl (0.4 g, 6.9 mmol) was added. The complex of aluminum chloride and NaCl was filtered off. The filtrate was distilled under vacuum to give (diphenylmethyl)methyldichlorosilane (**3b**, 3.2 g). Alternatively, the reaction of **2** and **1b** was carried out using 10 mol% AlCl_3 (0.2 g, 1.5 mmol) instead of 20 mol% under the same reaction condition above. **3b** (3.7 g) was obtained by the same procedure above. Spectral data for **3b**: $^1\text{H-NMR}$ (300 MHz) δ 0.85 (s, 3H, SiCH_3), 4.12 (s, 1H, CH), 7.33–7.51 (m, 10H, phenyl-*H*); $^{13}\text{C-NMR}$ (75 MHz) δ 4.81 (SiCH_3), 48.58 (SiCH), 126.58, 128.69,

129.22, 138.27 (phenyl-carbons). Mass spectrum: m/z (relative intensity) 284 (1), 282 (5), 280 (8) (M^+), 168 (14), 167 (100, Ph_2HC^+), 166 (11), 165 (34), 152 (15). For other physical properties of **3b**, refer to the previous report [14].

3.3. General procedure for the reaction of **2** with (dichloromethyl)silanes **1c** and **1d**

Using the procedure described in the reaction of **2** with **1a**, the reactions of **2** (11.7 g, 150.0 mmol), with (dichloromethyl)silanes **1c** (15.0 mmol) and **1d** (15.0 mmol) in the presence of aluminum chloride (0.4 g, 3.0 mmol) were carried out at r.t. or 80°C, respectively. The progresses of the reactions were monitored by GLC. The reaction results are summarized in Table 1. In the reaction with **1c**, (diphenylmethyl)dimethylchlorosilane (**3c**) was purified by a preparative GLC and characterized by NMR spectra. Spectral data for **3c**: $^1\text{H-NMR}$ (600 MHz) δ 0.44 (s, 6H, SiCH_3), 3.80 (s, 1H, CH), 7.18–7.35 (m, 10H, phenyl-*H*); $^{13}\text{C-NMR}$ (150 MHz) δ 1.45 (SiCH_3), 47.23 (SiCH), 125.94, 128.56, 129.05, 140.51 (phenyl-carbons). Mass spectrum: m/z (relative intensity) 262 (9), 260 (25) (M^+), 168 (15), 167 (100, Ph_2HC^+), 166 (10), 165 (29), 152 (14), 95 (12), 93 (34). For other properties of **3c**, refer to the previous report [15].

3.4. General procedure for the reaction of **2** with (trichloromethyl)silanes **1e** and **1f**

Using the procedure described in the reaction of **2** with **1a**, the reactions of **2** (11.7 g, 150.0 mmol) with (trichloromethyl)silanes **1e** (3.8 g, 15.0 mmol) and **1f** (3.5 g, 15.0 mmol) were carried out in the presence of aluminum chloride (0.4 g, 3.0 mmol) at r.t. or 80°C, respectively. The progresses of the reactions were monitored by GLC. After reactant **1** was consumed, NaCl (0.4 g, 6.9 mmol) was added. Then, the reaction mixture was stirred at reflux temperature of benzene for 2 h. The complex of aluminum chloride and NaCl was filtered off. The triphenylated products, (triphenylmethyl)trichlorosilane (**3e**) and (triphenylmethyl)methyldichlorosilane (**3f**), were isolated by recrystallization from toluene solution, respectively. Samples for melting points were further purified from toluene + THF solution. The reaction products are summarized in Table 2.

Data for **3e**: m.p. 209°C. $^1\text{H-NMR}$ (300 MHz) δ 7.27–7.39 (m, 15H, phenyl-*H*). $^{13}\text{C-NMR}$ (75 MHz) δ 60.29 (SiC), 127.32, 128.46, 130.42, 141.43 (phenyl-carbons). Mass spectrum: m/z (relative intensity) 244 (24), 243 (100, Ph_3C^+), 165 (41). Anal. Calc. for $\text{C}_{19}\text{H}_{15}\text{Cl}_3\text{Si}$: C, 60.41; H, 4.00. Found: C, 60.27; H, 4.08. Data for **3f**: mp. 194°C. $^1\text{H-NMR}$ (300 MHz) δ 0.90 (s, 3H, CH_3), 7.21–7.35 (m, 15H, phenyl-*H*).

^{13}C -NMR (75 MHz) δ 7.81 (SiCH_3), 57.48 (SiC), 126.78, 128.32, 130.33, 142.83 (phenyl-carbons). Mass spectrum: m/z (relative intensity) 358 (1), 356 (1) (M^+), 244 (22), 243 (100, Ph_3C^+), 165 (45). Anal. Calc. for $\text{C}_{20}\text{H}_{18}\text{Cl}_2\text{Si}$: C, 67.22; H, 5.08. Found: C, 66.98; H, 5.10.

3.5. Decomposition reaction of **3f** to **3b** in the presence of AlCl_3

The mixture of **3f** (0.5 g, 1.4 mmol), aluminum chloride (0.037g, 0.28 mmol), and dodecane (0.2 g) in benzene (2 ml) was stirred at r.t. and 80°C . The progress of the reactions was monitored by GLC. At r.t., the reaction did not occur. At 80°C , 15% of **3f** was decomposed to give **3b** (10%) for 1 h and the 28% consumption of **3f** gave **3b** (20%) for 2 h.

Acknowledgements

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