Reactions of methylchlorodisilanes with Grignard reagents

V. V. Semenov,* E. Yu. Ladilina, T. A. Chesnokova, N. K. Elistratova, Yu. A. Kurskii, and N. P. Makarenko

Institute of Organometallic Chemistry, Russian Academy of Sciences,
49 ul. Tropinina, 603137 Nizhnii Novgorod, Russian Federation.
Fax: +7 (831 2) 66 1497

Reactions of $Cl_2MeSiSiMeCl_2$ with RMgCl make it possible to obtain and isolate pure disilanes containing a smaller number of functional groups, namely, $RMeClSiSiMeCl_2$ (R = Ph), RMeClSiSiMeRCl (R = Pr^i, Ph), and $R_2MeSiSiMeRCl$ (R = Bu^i). The reaction of $Cl_2MeSiSiMeCl_2$ with Bu^nMgCl is the least selective. The chlorides obtained were reduced with $LiAlH_4$ into the corresponding hydrides.

Key words: organodisilanes, organohydrodisilanes, nucleophilic substitution; Grignard reagents.

Of the series of polychloro-substituted disilanes, $Si_2Me_nCl_{6-n}$, 1,2-dimethyl-1,1,2,2-tetrachlorodisilane (n = 2) is the most readily accessible compound. Significant amounts of this compound are found in the so-called disilane fraction (DSF),[1] which is isolated by rectification of the residues formed during industrial synthesis of organosilicon monomers. This fraction consists of two chloromethyldisilanes, $Si_2Me_2Cl_4$ and $Si_2Me_3Cl_3$ (molar ratio from 70 : 30 to 80 : 20).[1–5] These compounds can be used as starting reagents for syntheses of organodisilanes containing a smaller number of functional groups.

In the present work we report data on the reactions of 1,2-dimethyl-1,1,2,2-tetrachlorodisilane and DSF with Grignard reagents, RMgCl (R = Pr^i, Bu^n, Bu^i, Ph), and on the reduction of the resulting chlorides with lithium aluminumhydride.

Results and Discussion

We synthesized organochlorodisilanes, $Si_2Me_2R_nCl_{4-n}$, from alkyl and aryl chlorides (rather than from bromides). These halides were chosen because the reactions of RMgBr with silicon chlorides result in mixed organochlorobromosilanes, which complicates significantly the isolation of pure compounds. The reactions were carried out in THF or in a THF—heptane mixture with a molar ratio $Si_2Me_2Cl_4$: RMgCl = (1 : 0.5)—(1 : 4). It follows from the data in Table 1 that only reactions involving Pr^iMgCl and Bu^iMgCl occur with sufficient selectivity.

For example, the reaction of Pr^iMgCl (2 mol) with $Si_2Me_2Cl_4$ (1 mol) results in 1,2-dimethyl-1,2-diisopropyl-1,2-dichlorodisilane (**1**), which can be isolated in the pure form, as the main product.[6] Prolonged heating

Table 1. Conditions of the reactions of $Cl_2MeSiSiMeCl_2$ with Grignard reagents and yields of substitution products

RMgX	RMgX : $Si_2Me_2Cl_4$[a]	Solvent	T/°C	Reaction time/h	Products (yield (%))[b] $Si_2Me_2RCl_3$	$Si_2Me_2R_2Cl_2$	$Si_2Me_2R_3Cl$	$Si_2Me_2R_4$
Pr^iMgCl	2:1	THF	65—70	2	**3** (8)	**1** (92)		
	3:1	THF	65—70	21		**1** (72)	**2** (28)	
Bu^nMgCl	3:1	THF	35—40	4		**6** (10)	**7** (65)	**8** (25)
	4:1	THF	35—40	4			**7** (4)	**8** (96)
Bu^iMgCl	3:1	THF	65—70	7			**9** (98)	
PhMgCl	0.5:1	THF—heptane	20—25	48	**11** (50)			
	1:1	THF—heptane	20—25	48	**11** (98)	**12** (2)		
	2:1	THF—heptane	20—25	48	**11** (35)	**12** (44)	**13** (21)	
	3:1	THF—heptane	20—25	48	**11** (9)	**12** (13)	**13** (78)	
PhMgBr	4:1	THF	65—70	10				**14** (84)[c]
	4:1	Ether	36—40	10				**14** (11)[c]

[a] Molar ratio. [b] GLC data. [c] After purification.

Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 5, pp. 954—957, May, 1995.

of a mixture of Pr^iMgCl (3 mol) and $Si_2Me_2Cl_4$ (1 mol) results in slow accumulation of trisubstituted product **2**. After 21 h, the ratio of compounds **1** : **2** was 72 : 28. We were unable to isolate compound **2** from a mixture with **1** by rectification.

The reaction of Pr^iMgCl (2 mol) with DSF (0.78 mol $Si_2Me_2Cl_4$ and 0.22 mol $Si_2Me_3Cl_3$) gave dichlorodisilane **1**, 1,2-dimethyl-1-isopropyl-1,2,2-trichlorodisilane (**3**), and a mixture of two isomeric disilanes with the general formula $Si_2Me_3Pr^iCl_2$ (**4a**,**b**).

$$Si_2Me_2Cl_4/\ Si_2Me_3Cl_3 \xrightarrow[THF]{Pr^iMgCl} \underset{\mathbf{1}}{Pr^iMeClSiSiMeClPr^i} +$$

$$+ \underset{\mathbf{3}}{Pr^iMeClSiSiMeCl_2} + \underset{\mathbf{4a}}{Pr^iMeClSiSiMe_2Cl} + \underset{\mathbf{4b}}{Pr^iMe_2SiSiMeCl_2}$$

Rectification of the reaction mixture gave compound **1** and a mixture of isomers **4a**,**b**.

The reactions involving *n*-butylmagnesium chloride were found to be the least selective. For example, the reaction of $Si_2Me_2Cl_4$ (1 mol) with Bu^nMgCl (3 mol) resulted in products of the replacement of two, three, and four Cl atoms. Five minutes after the reagents were mixed, the reaction mixture contained the completely substituted disilane **8** (GLC data, see Table 1), yield 16 %. GLC was unable to detect the presence of the monobutyl derivative, $Si_2Me_2BuCl_3$. These data show that the reaction of $Si_2Me_2Cl_4$ with Bu^nMgCl occurs readily. Even when three *n*-butyl groups are introduced, no noticeable steric hindrance is created, and the replacement of the remaining Cl atom does not meet substantial difficulties. The completely substituted disilane **8** is readily formed in the reaction of $Si_2Me_2Cl_4$ (1 mol) with Bu^nMgCl (4 mol).

The reaction involving Bu^iMgCl (unlike that with the *n*-butyl Grignard reagent) was found to be highy selective. Using the ratio $Si_2Me_2Cl_4$: Bu^iMgCl = 1 : 3, we obtained the expected 1,2-dimethyl-1,1,2-triisobutyl-2-chlorodisilane (**9**, see Table 1). GLC did not detect the high-boiling product of complete substitution, $Si_2Me_2Bu^i_4$, in the reaction mixture. This indicates that isobutyl groups ($E_s = -0.405$) create considerably higher steric hindrance against the introduction of the fourth substituent than *n*-butyl groups ($E_s = -0.225$).[7]

The reactions of phenylmagnesium chloride with $Si_2Me_2Cl_4$ in THF result in complex mixtures of compounds with different numbers of substituents. The selectivity increases if a solution of PhMgCl in THF is added to a solution of the chlorosilane in heptane. However, only the mono-substituted disilane **11** can be obtained in a high yield using this method (see Table 1). When the $Si_2Me_2Cl_4$: PhMgCl ratio was 1 : 2 or 1 : 3, mono-, di-, and trisubstituted products were formed simultaneously. The boiling points differ considerably, which makes it possible to isolate pure 1,2-dimethyl-1-phenyl-1,2,2-trichlorodisilane (**11**)[8] and 1,2-dimethyl-1,2-diphenyl-1,2-dichlorodisilane (**12**). 1,2-Dimethyl-1,1,2-triphenyl-2-chlorodisilane (**13**) could be obtained more conveniently by controlled chlorodephenylation of 1,2-dimethyl-1,1,2,2-tetraphenyldisilane (**14**).

$$\underset{\mathbf{14}}{Ph_2MeSiSiMePh_2} + HCl \xrightarrow[CHCl_3]{AlCl_3} \underset{\mathbf{13}}{Ph_2MeSiSiMePhCl} + PhH$$

Exhaustive phenylation of $Si_2Me_2Cl_4$ with phenylmagnesium bromide in ether gives tetra-substituted disilane **14** in an unexpectedly low yield (11 %). The target product is strongly contaminated with diphenyl. The use of THF makes it possible to increase the yield to 84 %.

Organochlorodisilanes are reduced with $LiAlH_4$ in ether to give the corresponding hydrides, namely, $Pr^iMeHSiSiHMePr^i$ (**5**), $Bu^i_2MeSiSiHMeBu^i$ (**10**), and $Ph_2MeSiSiMePhH$ (**15**).

The IR spectra of the resulting chloromethylphenyldisilanes **11**—**14** contain bands typical of Si—Ph, Si—Me, and Si—Cl fragments.[9,10] The absorption bands at 3060—3040 cm^{-1} corresponding to the stretching vibrations, νCH, of the benzene ring, are more intense than similar νCH bands of the methyl groups (2940 cm^{-1}). When the number of phenyl groups in the disilane increases, the ratio of the intensities of νCH(Ph)/νCH(Me) increases. The stretching vibrations of the aromatic skeleton, νCC, appear as three bands at 1580, 1475, and 1420 cm^{-1}. The latter band is particularly intense in the spectra of all of the compounds. Due to the presence of phenyl groups, a very intense band is also observed at 1100 cm^{-1}. The doublet or triplet at 690—740 cm^{-1} can be attributed to the out-of-plane deformation vibrations of the C—H bond in the benzene ring, and the two bands at 750—800 cm^{-1} can be attributed to similar ρCH_3 vibrations. The σCH planar deformation vibrations of the phenyl groups are represented by two low-intensity bands at 1330 and 1290 cm^{-1}. The presence of Si—Me fragments is most clearly demonstrated by the strong band at 1240—1245 cm^{-1} typical of symmetric δ_s deformation vibrations of Me groups. In the spectrum of 1,2-dimethyl-1,1,2,2-tetraphenyldisilane this band has medium intensity. The introduction of Cl atoms into $Si_2Me_2Ph_4$ instead of phenyl groups is accompanied by the appearance of new absorption bands of medium or high intensity at 450—550 cm^{-1}. The IR spectrum of hydrodisilane **15** contains two high-intensity bands at 2080 and 880 cm^{-1} corresponding to the stretching and deformation vibrations of the Si—H bond, respectively.

The ^1H NMR spectrum of 1,2-dimethyl-1,2-diphenyl-1,2-dichlorodisilane **12** displays the resonances of methyl group protons as two lines with a 1 : 1 intensity ratio ($\Delta\delta = 0.06$). The appearance of two resonances with low Δδ results from the presence of isomers of disilane **12**. A molecule of **12** has two asymmetric Si atoms linked with similar substituents, *i.e.*, it looks like an analog of the classical dihydroxysuccinic acids that exist as D-, L-, and *meso*-forms. It is known from the literature data[11,12] for the carbon analogs, the substituted butanes MeCHXCHXMe (X = Cl, Br, Ph), that the chemical shifts for D- and L-isomers are the same, while the signal of the *meso*-form is shifted 0.04—0.25 ppm upfield relative to that of the D,L-form.

Experimental

GLC analyses of organodisilanes and reaction mixtures were carried out on a Tsvet-530 gas chromatograph (a 0.3×200 cm stainless steel column packed with Chromaton N-AW-DMCs with 5 % SE-30 silicone, a thermal conductivity detector, and helium as the carrier gas). Phenyl-substituted disilanes were analyzed by GLC (Tsvet-530) and HPLC (Millikhrom-1A chromatograph, 0.2×6.4 cm column packed with 5 μm Silasorb-600, UV detector (250 nm), and heptane—THF (400 : 1) as the eluent, v = 200 μL min^{-1}). Retention times increased in the series: diphenyl < **13** < **14** < **12** < **11**.

IR spectra were recorded on a Perkin-Elmer-577 spectrophotometer. ^{1}H NMR spectra were recorded on a Tesla BS-567A spectrometer (100 MHz) using $CDCl_3$ as the solvent and $CHCl_3$ as the internal standard (δ 7.25).

The solvents were purified according to standard procedures.[13] The disilane fraction (150—160 °C) was isolated by rectifying the methyl residues. 1,2-Dimethyl-1,1,2,2-tetrachlorodisilane was obtained from DSF according to the reported procedure.[4]

The reaction of isopropylmagnesium chloride with DSF. A solution of DSF (285.0 g; 1.0 mol of $Si_2Me_2Cl_4$ and 0.28 mol of $Si_2Me_3Cl_3$) in THF (300 mL) was added with stirring and cooling (ice water) to the Grignard reagent obtained from Mg (60.0 g, 2.5 g-at) and Pr^{i}Cl (206.3 g, 2.5 mol) in THF (1100 mL). The reaction mixture was refluxed for 7 h and filtered. The filtrate was concentrated, hexane (600 mL) was added to the residue, and the mixture was filtered once more. The solvent was distilled off, and the residue was fractionated two times *in vacuo* with a dephlegmator to give 121.5 g (50 %) of compound **1** (b.p. 90—92 °C (5 Torr), n_D^{20} 1.4751), 30.5 g of a mixture of compounds **3** and **4**, and 23.7 g (39 %) of a mixture of isomers **4a,b** (3 : 4, b.p. 75—80 °C (5 Torr), n_D^{20} 1.4690, d_4^{20} 1.0330, MR_D 58.03 (calcd. 59.06)). Found (%): C, 33.41; H, 7.87; Cl, 34.16; Si, 25.68. $C_6H_{16}Cl_2Si_2$. Calculated (%): C, 33.48; H, 7.48; Cl, 32.94; Si, 26.09. ^{1}H NMR ($CHCl_3$), δ: compound **3**: 0.938 (s, 3 H, $MeSiCl_2$), 0.578 (s, 3 H, MeSiPr^{i}Cl); compound **4a**: 0.500 (s, 3 H, MeSiPr^{i}Cl), 0.593 (s, 6 H, Me_2SiCl); compound **4b**: 0.841 (s, 3 H, $MeSiCl_2$), 0.198 (s, 6 H, Me_2SiPr^i), 1.05—1.20 (m, Me_2CHSi).

1,2-Dimethyl-1,2-diisopropyldisilane (5). Dichlorodisilane **1** (120.0 g, 0.49 mol) in ether (100 mL) was added with stirring to a solution of $LiAlH_4$ (38.0 g, 1 mol) in ether (800 mL), and the reaction mixture was refluxed for 2 h. The excess $LiAlH_4$ was decomposed with distilled water, and the ethereal solution was dried with Na_2SO_4. The ether was distilled off, and the residue was fractionated under ambient pressure in a slow stream of argon to give 38.2 g (45 %) of compound **5**, b.p. 175—178 °C, n_D^{20} 1.4535, d_4^{20} 0.7770, MR_D 60.75 (calcd. 61.01). Found (%): C, 55.18; H, 12.87; Si, 31.60. $C_8H_{22}Si_2$. Calculated (%): C, 55.09; H, 12.71; Si, 32.20. IR, ν/cm^{-1}: 2960 vs, 2945 vs, 2895 m, 2870 vs (C—H); 2090 vs (Si—H); 1465 s, 1250 s (Si—C); 1080 w, 1010 m, 940 w, 875 vs, 790 vs, 782 vs, 735 m, 700 m.

1,2-Dimethyl-1,1,2,2-tetrabutyldisilane (8). $Si_2Me_2Cl_4$ (45.0 g, 0.20 mol) in THF (50 mL) was added dropwise with stirring to the Grignard reagent obtained from Mg (24.3 g, 1.0 g-at) and BuCl (92.6 g, 1.0 mol) in THF (400 mL). Mixing was accompanied by self-heating of the mixture. GLC analysis showed that the $Si_2Me_2Cl_4$ was completely transformed into $Si_2Me_2Bu_4$. The excess BuMgCl was decomposed with water, then hexane (200 mL) was added. The organic layer was separated and dried with $CaCl_2$, and concentrated. The residue was fractionated *in vacuo* to give 38.4 g (62 %) of compound **8**, b.p. 132—134 °C (1 Torr), n_D^{20} 1.4650, d_4^{20} 0.8180, MR_D 106.35 (calcd. 106.95). Found (%): C, 68.52; H, 13.58; Si, 17.15. $C_{18}H_{42}Si_2$. Calculated (%): C, 68.70; H, 13.45; Si, 17.85. IR, ν/cm^{-1}: 2940 vs, 2900 vs, 2850 vs, 1450 vs, 1400 m, 1370 s, 1330 m, 1285 m, 1240 vs, 1180 m, 1170 m, 1075 s, 1020 w, 995 m, 960 m, 870 s, 790 s, 770 vs, 725 s.

1,2-Dimethyl-1,1,2-triisobutyl-2-chlorodisilane (9). $Si_2Me_2Cl_4$ (76.0 g, 0.33 mol) in THF (100 mL) was added with stirring and cooling with ice water to the Grignard reagent obtained from Mg (24.3 g, 1.0 g-at) and Bu^{i}Cl (92.6 g, 1.0 mol) in THF (400 mL). The reaction mixture was refluxed for 7 h and filtered. The filtrate was concentrated and distilled *in vacuo*. Repeated distillation gave 60.8 g (63 %) of compound **9**, b.p. 130—135 °C (0.1 Torr), n_D^{20} 1.4690, d_4^{20} 0.8980, MR_D 90.88 (calcd. 92.57). Found (%): C, 57.16; H, 11.52; Cl, 12.22; Si, 19.30. $C_{14}H_{33}ClSi_2$. Calculated (%): C, 57.38; H, 11.35; Cl, 12.10; Si, 19.17. IR, ν/cm^{-1}: 2940 vs, 2880 vs, 2860 vs, 1455 vs, 1395 m, 1375 m, 1360 s, 1320 s, 1245 vs, 1215 s, 1160 s, 1085 s, 1030 s, 830 s, 795 s, 770 vs, 500 s, 460 m, 425 w.

1,2-Dimethyl-1,1,2-triisobutyldisilane (10). Dichlorodisilane **9** (29.0 g, 0.1 mol) in ether (30 mL) was added with stirring to $LiAlH_4$ (3.8 g, 0.1 mol) in ether (100 mL), and the reaction mixture was refluxed for 2 h. The excess $LiAlH_4$ was decomposed with water, and the ethereal solution was dried with Na_2SO_4. The ether was distilled off, and the residue was fractionated *in vacuo* to give 16.2 g (62 %) of compound **10**, b.p. 120—122 °C (0.1 Torr), n_D^{20} 1.4626, d_4^{20} 0.8080, MR_D 88.07 (calcd. 88.63). Found (%): C, 64.61; H, 13.24; Si, 21.18. $C_{14}H_{34}Si_2$. Calculated (%): C, 65.07; H, 13.24; Si, 21.72. IR, ν/cm^{-1}: 2080, 875 (Si—H).

The reaction of phenylmagnesium chloride with 1,2-dimethyl-1,1,2,2-tetrachlorodisilane. A solution of PhMgCl in THF (485 mL, 1.86 mol L^{-1}, 0.9 mol) was added over a period of 2.5 h with stirring and cooling (ice water) to $Si_2Me_2Cl_4$ (103.0 g, 0.45 mol) in heptane (420 mL). The flask was heated for 10 h at boiling points of the solvents. The reaction mixture was filtered, the precipitate was washed with heptane, and the filtrate was concentrated and distilled *in vacuo* and six fractions with b.p. from 83 to 225 °C (0.5 Torr) were collected. Subsequent repeated rectification gave 29.6 g (24.4 %) of disilane **11**, 32.9 g (24.4 %) of compound **12**, and 7.9 g (4.4 %) of compound **13**. Compounds **11** and **12** were pure, while product **13** contained an admixture of diphenyl and disilanes **12** and **14**. 1,2-Dimethyl-1-phenyl-1,2,2-trichlorodisilane (**11**), b.p. 111—112 °C (1 Torr). Found (%): C, 36.38; H, 4.35; Cl, 38.81; Si, 20.66. $C_8H_{11}Cl_3Si_2$. Calculated (%): C, 35.62; H, 4.11; Cl, 39.40; Si, 20.83. ^{1}H NMR (CH_2Cl_2), δ: 1.00 (s, 3 H, SiMeClPh); 1.02 (s, 3 H, $SiMeCl_2$); 7.4—7.9 (m, 5 H, Ph). 1,2-Dimethyl-1,2-diphenyl-1,2-dichlorodisilane (**12**) (D,L- and *meso*-forms), b.p. 185—186 °C (2 Torr). Found (%): C, 54.58; H, 5.79; Cl, 22.80; Si, 17.91. $C_{14}H_{16}Cl_2Si_2$. Calculated (%): C, 54.00; H, 5.18; Cl, 22.77; Si, 18.04. ^{1}H NMR, δ: 0.96 (s, 3 H, SiMe, *meso*-form); 1.02 (s, 3 H, SiMe, D,L-forms); 7.4—7.9 (m, 10 H, Ph).

1,2-Dimethyl-1,1,2-2-tetraphenyldisilane (14). $Si_2Me_2Cl_4$ (68.0 g, 0.3 mol) in ether (400 mL) was added with stirring and cooling to the Grignard reagent obtained from Mg (36.0 g, 1.5 g-at) and PhBr (235.5 g, 1.5 mol) in ether (550 mL). The mixture was heated for 10 h and cooled, then dilute HCl (400 mL) was added. The organic layer was washed with distilled water, dried with $CaCl_2$, and concentrated. The residue (viscous liquid) was heated for 2 h at 150 °C *in vacuo*. A colorless transparent liquid (4.1 g) and diphenyl (2.3 g) were distilled off. The remaining oil was dissolved in heated ethanol. Cooling gave crystals of **14** contaminated with diphenyl (HPLC data). Repeated recrystallization from ethanol or heptane gave 12.5 g (10.6 %) of pure disilane **14**, m.p. 142—143 °C (Ref. 14: 142—143 °C). Found (%): C, 78.72; H, 6.69; Si, 13.86. $C_{26}H_{26}Si_2$. Calculated (%): C, 79.13; H, 6.64; Si, 14.23. ^1H NMR, δ: 0.76 (s, 6 H, SiMe, $^2J_{^1H-^{29}Si}$ = 6.29 Hz); 7.25—7.55 (m, 20 H, Ph).

1,2-Dimethyl-1,1,2-triphenyl-2-chlorodisilane (13). Dry HCl was passed with stirring through a mixture of disilane **14** (5.36 g, 0.014 mol) and $AlCl_3$ (0.28 g, 0.002 mol) in $CHCl_3$ (100 mL). Samples for GLC analysis were withdrawn at regular intervals. After 7 h, the appearance of dichloride **12** was detected. The HCl flow was stopped, and the reaction mixture was filtered and concentrated *in vacuo*. Heptane (20 mL) was added to the residue. After 20 h, the $AlCl_3$ and the original disilane **14** that precipitated were filtered off. The solvent was distilled off, and the residue was distilled *in vacuo* to give 2.40 g (50 %) of disilane **13**, b.p. 233—235 °C (1 Torr). Found (%): C, 68.15; H, 6.60; Si, 16.01; Cl, 10.16. $C_{20}H_{21}ClSi_2$. Calculated (%): C, 68.05; H, 6.00; Si, 15.91; Cl, 10.04. ^1H NMR, δ: 0.87 (s, 3 H, $Si\underline{Me}Ph_2$, $^2J_{^1H-^{29}Si}$ = 6.76 Hz, $^3J_{^1H-^{29}Si}$ = 3.42 Hz); 0.89 (s, 3 H, $Si\underline{Me}PhCl$, $^2J_{^1H-^{29}Si}$ = 6.29 Hz, $^3J_{^1H-^{29}Si}$ = 1.78 Hz); 7.25—7.80 (m, 15 H, Ph).

1,2-Dimethyl-1,1,2-triphenyldisilane (15) was obtained similarly to compound **10** from chlorodisilane **13** and $LiAlH_4$, b.p. 80—82 °C (0.3 Torr), n_D^{20} 1.6073. Found (%): C, 75.14; H, 7.05; Si, 17.67. $C_{20}H_{22}Si_2$. Calculated (%): C, 75.41; H, 6.96; Si, 17.63.

References

1. R. Calas, J. Dunogues, G. Deleris, and N. Duffaut, *J. Organomet. Chem.*, 1982, **225**, 117.
2. A. N. Polivanov, N. N. Troitskaya, E. V. Los', N. N. Silkina, A. S. Shapatin, M. P. Gavars, O. D. Gracheva, A. G. Trufanov, and V. N. Bochkarev, *Zh. Obshch. Khim.*, 1987, **57**, 1570 [*J. Gen. Chem. USSR*, 1987, **57** (Engl. Transl.)].
3. J. Cermak and J. Franc, *Coll. Czechosl. Chem. Commun.*, 1965, **30**, 3278.
4. H. Matsumoto, T. Motegi, M. Hasagawa, and J. Nagai, *J. Organomet. Chem.*, 1977, **142**, 149.
5. R. Lehnert, M. Hoppner, and H. Kelling, *Z. Anorg. Allgem. Chem.*, 1990, **591**, 209.
6. T. A. Chesnokova and V. V. Semenov, *Metalloorg. Khim.*, 1990, **3**, 629 [*Organomet. Chem. USSR*, 1990, **3**, 320 (Engl. Transl.)].
7. F. K. Cartledge, *Organometallics*, 1983, **2**, 425.
8. J. Grobe, G. Henkel, B. Krebs, and N. Voulgarakis, *Z. Naturforsch.*, 1984, **39b**, 341.
9. M. A. Chumaevskii, in *Kolebatel'nye spektry elementoorganicheskikh soedinenii elementov IVB i VB grupp* [*Vibrational Spectra of Group IVB and VB Elements*], Nauka, Moscow, 1971, 49 (in Russian).
10. V. G. Osipov, N. N. Silkina, G. G. Baukova, and E. A. Chernyshev, in *Spektry i khromatogrammy elementoorganicheskikh soedinenii. Vyp. 1. IK- i UF-spektry fenilsilanov* [*Spectra and Chromatograms of Organometallic Compounds. Issue 1. IR and UV Spectra of Phenylsilanes*], Khimiya, Moscow, 1976, 3 (in Russian).
11. A. A. Bothner-By and C. Naar-Colin, *J. Am. Chem. Soc.*, 1962, **84**, 743.
12. F. A. L. Anet, *J. Am. Chem. Soc.*, 1962, **84**, 747.
13. A. Weisberger, E. Proskauer, J. Riddic, and E. Tups, in *Organic Solvents*, Plenum, New York, 1955.
14. O. W. Steudel and H. Gilman, *J. Am. Chem. Soc.*, 1960, **82**, 6129.

Received February 23, 1994;
in revised form December 14, 1994