

Synthesis and Properties of 1,2-Dichlorodisilane and 1,1,2-Trichlorodisilane

Andreas Gupper^[a] and Karl Hassler^{*[a]}

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The previously unknown disilanes 1,1,2-trichlorotri(1-naphthyl)disilane and 1,2-dichlorotetra(1-naphthyl)disilane were prepared by the reaction of 1-lithionaphthalenide with hexachlorodisilane. Reduction with LiAlH_4 afforded 1,1,2-tri(1-naphthyl)disilane. Treatment of 1,2-di(1-naphthyl)disilane and 1,1,2-tri(1-naphthyl)disilane with liquid hydrogen chloride gave $\text{ClH}_2\text{SiSiH}_2\text{Cl}$ and $\text{ClH}_2\text{SiSiHCl}_2$ in quantitative yields, along with naphthalene. The chlorodisilanes were easily separated and purified by fractional condensation.

Both disilanes are stable for weeks at room temperature, but undergo rapid H/Cl exchange reactions and SiSi cleavage upon the addition of traces of catalysts such as AlCl_3 . The ^{29}Si chemical shifts and $J(^{29}\text{Si}, ^1\text{H})$ coupling constants, including those of $\text{ClH}_2\text{SiSiH}_3$ and $\text{Cl}_2\text{HSiSiH}_3$, are reported. The infrared and Raman vibrational spectra clearly prove that both chlorodisilanes comprise mixtures of *gauche* and *anti* rotamers in the liquid state.

Introduction

The chemical properties of hydridohalosilanes $\text{Si}_2\text{H}_n\text{X}_{6-n}$ ($\text{X} = \text{F}, \text{Cl}, \text{Br}$ and I) differ considerably from those of their carbon analogues $\text{C}_2\text{H}_n\text{X}_{6-n}$. First, the polarities of the SiH and CH bonds are opposite ($\text{Si}^{\delta+}\text{H}^{\delta-}$ vs. $\text{C}^{\delta-}\text{H}^{\delta+}$), and second the silicon atom easily forms transition states with coordination numbers >4 because of its larger size. Therefore, hydridohalosilanes easily react with water forming Si–O–Si linkages and hydrogen halides HX. In contrast to the halogenated ethanes, the activation energy for the reaction of halosilanes with oxygen is very low, making them spontaneously inflammable on contact with air. In addition, they are very prone to undergo halogen/hydrogen exchange reactions, which is not the case for the haloethanes.

The combination of these properties makes the synthesis and the handling of the compounds a difficult task. Earlier attempts focused on the reaction of disilane Si_2H_6 with various halogenating agents. For chlorodisilanes, HCl ,^[1–3] Cl_2 ,^[4] BCl_3 ,^[5–8] AgCl ^[3] or SnCl_4 ^[9] were used. Not unexpectedly, mixtures of chlorodisilanes were obtained that were difficult or even impossible to separate. Methods which avoided Si_2H_6 as a starting material comprised the reaction of monoiododisilane with AgCl ,^[10] the reaction of SiCl_2H_2 in an electrical discharge^[11,12] or the electrophilic cleavage of the Si-phenyl bonds of tetraphenyldisilane (and other phenyldisilanes) with HCl/AlCl_3 .^[13] By these procedures, only $\text{Si}_2\text{H}_5\text{Cl}$ could be obtained in pure form and in preparative quantities. The reaction of tetraphenyldisilane with HCl/AlCl_3 ^[13] had the disadvantage that the benzene which formed simultaneously could not be separated from the tetrachlorodisilane. In 1979, the isolation of pentachlorodisilane from a mixture of chlorosilanes obtained as a by-

product of the thermal decomposition of HSiCl_3 for the generation of ultrapure semiconductor-grade silicon was reported.^[14]

HBr and HI ,^[15,2,16] Br_2 ^[4] and I_2 ,^[17,18] as well as BBr_3 and BI_3 ,^[8,19] have all been used for the bromination or iodination of disilane. Monobromodisilane and monoiododisilane could be successfully prepared and purified, but not disilanes with a number of halogen atoms exceeding one.

Synthetic strategies for the preparation of fluorodisilanes comprised reactions of difluorosilylene SiF_2 with H_2S ,^[20] B_2H_6 ,^[21] HBr ,^[22] or PH_3 ,^[23] as well as the selective reduction of bromofluorodisilanes such as $\text{F}_3\text{SiSiHBr}_2$ with Me_3SnH ^[24] or halogen exchange with SbF_3 ^[8] or ZnF_2 .^[25] So far, FSi_2H_5 ,^[26] F_3SiSiH_3 ,^[21,24] $\text{F}_5\text{Si}_2\text{H}$ ^[22] and $\text{F}_2\text{HSiSiHF}_2$ ^[25] have been synthesised by these procedures.

In our group we have focused on the electrophilic cleavage of silicon-aryl bonds by HCl , HBr or HI :



Under appropriate conditions, SiH functionalities are not attacked and the method can be used for the synthesis of a variety of bromo- and iododisilanes $\text{X}_n\text{Si}_2\text{H}_{6-n}$,^[27] as well as bromo- and iodotrisilanes $\text{X}_n\text{Si}_3\text{H}_{8-n}$.^[28] If the boiling points of the disilanes and trisilanes differ considerably from that of benzene, hydridophenyldisilanes and -trisilanes can be used as starting materials as their synthesis poses the least problems.^[29] In all other cases, the introduction of larger aryl groups such as mesityl or naphthyl seems indispensable. For instance, 1,1,2,2-tetrachlorodisilane can be prepared from 1,1,2,2-tetramesityldisilane,^[25] and 1,2-dibromodisilane from 1,2-di(1-naphthyl)disilane.^[30] The disilanes can then be purified simply by distillation or fractional condensation. The disadvantage with this approach is that for the preparation of naphthyldisilanes and naphthyltrisilanes the coupling reaction of chloronaphthylmonosilanes with alkali metals is not an appropriate method due to side

^[a] Institute of Inorganic Chemistry, Technical University, Stremayrgasse 16, 8010 Graz, Austria
Fax: (internat.) +43-316/873-8701
E-mail: hassler@anorg.tu-graz.ac.at

reactions at the aryl group. Similar problems are encountered with mesityl groups due to their increased bulkiness.

In a previous paper we reported on the synthesis of pentachloro(1-naphthyl)disilane and tetrachloro-1,2-di(1-naphthyl)disilane from hexachlorodisilane and naphthyllithium.^[31] This method can be expanded for the preparation of tri- and tetranaphthylidisilanes as well, which in turn can be used for the preparation of the title hydrido-chlorodisilanes.

Results and Discussion

(1-Naphthyl)disilanes

The two most convenient methods for the preparation of arylated disilanes are the reductive coupling of the appropriate arylhalomonosilanes with Li, Na, K and Mg and the reaction of silyllithium, -sodium or -potassium compounds with arylhalosilanes. Unfortunately, neither method can be used for the synthesis of naphthylidisilanes as the naphthyl group easily reacts with alkali metals leading to a large number of badly defined products.

These difficulties can be circumvented by the use of naphthyllithium which, under appropriate conditions, reacts with hexachlorodisilane (although not without appreciable Si–Si bond cleavage) to form naphthylpentachlorodisilane and 1,2-dinaphthyltetrachlorodisilane:^[31]



Increasing the stoichiometric ratio of $\text{LiC}_{10}\text{H}_7$ vs. Si_2Cl_6 allows the preparation of 1,1,2-trichlorotrinaphthylidisilane and 1,2-dichlorotetranaphthylidisilane in moderate yields. Appreciable quantities of trichloronaphthylsilane are formed as a by-product due to increased Si–Si bond cleavage by $\text{LiC}_{10}\text{H}_7$. Both disilanes can be separated from the reaction mixture by distillation and can be purified by fractional crystallization. They are easily reduced with lithium aluminum hydride to give the corresponding naphthylhydridosilanes in nearly quantitative yields.

Chlorodisilanes

The arylated disilanes $\text{Ar}_n\text{Si}_2\text{H}_{6-n}$ are the most suitable precursors for the preparation of the halodisilanes $\text{X}_n\text{Si}_2\text{H}_{6-n}$ as the aryl groups are easily replaced by halogen atoms X (X = Cl, Br and I) upon reaction with either the liquefied hydrogen halides HX or solutions of HX in benzene. The latter method has the disadvantage that small quantities of AlCl_3 have to be used as a catalyst and this cannot usually be removed completely from the halodisilane. Furthermore, traces of AlCl_3 also catalyse halogen/hydrogen exchange reactions and Si–Si bond cleavage, thereby leading to a rapid decomposition of the disilanes. With iodine and bromine these reactions are quite slow and the disilanes can be stored at -70°C for several weeks

without any appreciable decomposition. For chlorodisilanes, disproportionation occurs quickly. Therefore, the chlorodisilanes described herein were prepared by the reaction of the naphthylidisilanes with liquefied HCl in a sealed tube. For instance, $\text{ClH}_2\text{SiSiH}_2\text{Cl}$ prepared this way can be stored for weeks at room temperature without any decomposition.

²⁹Si NMR spectra

Table 1 and 2 summarize the ²⁹Si chemical shifts (ppm against tetramethylsilane) and $J(^{29}\text{Si},^1\text{H})$ coupling constants (Hz) for the naphthylidisilanes and chlorodisilanes, respectively. The data for 1,1,1,3-tetrachlorodisilane and 1,1-dichlorodisilane have been included as these molecules were also identified in the reaction mixtures. Chlorodisilane $\text{ClH}_2\text{SiSiH}_3$ was also prepared for NMR spectroscopic purposes; the chemical shifts and coupling constants for related disilanes described elsewhere have been included, too.

Table 1. ²⁹Si NMR chemical shifts (δ_{Si} vs. TMS) and $J(^{29}\text{Si},^1\text{H})$ coupling constants (Hz) of (1-naphthyl)chlorodisilanes and (1-naphthyl)disilanes

Disilane	δ_{Si}	$\delta_{\text{Si}'}$	$^1J_{\text{SiH}}$	$^1J_{\text{Si'H}}$	$^3J_{\text{SiH}}$	$^3J_{\text{Si'H}}$
NaphCl ₂ SiSi'Cl ₃ ^[a]	1.8	0.6			9.6	
Naph ₂ ClSiSi'Cl ₃	−1.5	7.6			7.8	
NaphCl ₂ SiSiCl ₂ Naph ^[a]	6.3				9.1	
Naph ₂ ClSiSi'Cl ₂ Naph	−0.5	11.8			8.7	10.8
Naph ₂ ClSiSiClNaph ₂	1.2				8.0	
NaphH ₂ SiSi'H ₃ ^[a]	−65.8	−99.4	196.2	195.7		
Naph ₂ HSiSi'H ₃	−40.9	−97.6	189.7	193.0	8.9	6.0
NaphH ₂ SiSiH ₂ Naph ^[a]	−63.4		194.2		7.2	
Naph ₂ HSiSi'H ₂ Naph	−39.9	−61.7	190.4	191.9	7.0	7.9

[a] Taken from ref.^[31]

Table 2. ²⁹Si NMR chemical shifts (δ_{Si} vs. TMS) and $J(^{29}\text{Si},^1\text{H})$ coupling constants (Hz) of chlorodisilanes

Disilane	δ_{Si}	$\delta_{\text{Si}'}$	$^1J_{\text{SiH}}$	$^1J_{\text{Si'H}}$	$^2J_{\text{SiH}}$	$^2J_{\text{Si'H}}$
H ₃ SiSi'H ₂ Cl	−95.1	−27.9	203.7	226.3	12.8	6.6
H ₃ SiSi'HCl ₂	−88.9	8.3	210.5	267.3	23.4	8.9
ClH ₂ SiSiH ₂ Cl	−30.3		233.4		15.8	
Cl ₂ HSiSi'H ₂ Cl	−0.2	−32.1	277.7	242.2	19.8	29.3
Cl ₂ HSiSiHCl ₂ ^[a]	−5.9		287.2		36.7	
Cl ₃ SiSi'HCl ₂ ^[b]	−2.2	−8.2		294.0	45.6	
Cl ₃ SiSiCl ₃ ^[c]	−6.1					

[a] Taken from ref.^[25]. — [b] Taken from ref.^[13] — [c] Taken from ref.^[32]

Figure 1 displays the proton coupled ²⁹Si NMR spectra of chlorodisilane and 1,2-dichlorodisilane exemplifying the quality of the spectra that have been obtained. The inter-

pretation of the spectra is straightforward as all couplings are first order.

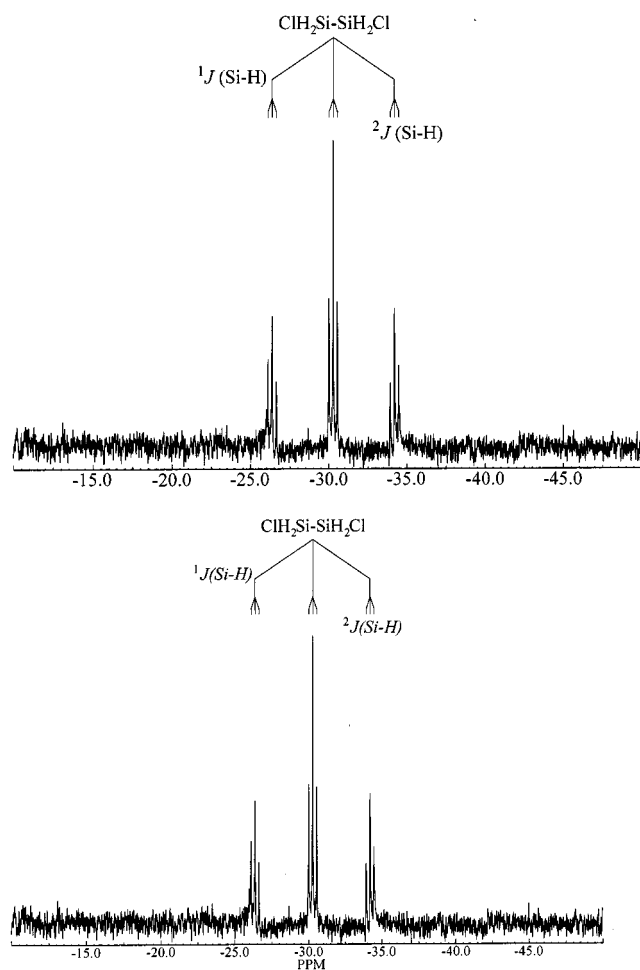


Figure 1. Proton coupled ^{29}Si NMR spectra of 1,2-dichlorodisilane (top) and chlorodisilane (bottom)

Figure 2 presents the spectrum of 1,1,2-trichlorodisilane in the range $\delta = +10$ to -40 after equilibration upon addition of AlCl_3 . Interestingly, no 1,2-dichlorodisilane is formed. 1,1-Dichlorodisilane and 1,1,2,2-tetrachlorodisilane are readily detected, along with monosilanes such as H_3SiCl , H_2SiCl_2 and HSiCl_3 .

From mixtures of chlorodisilanes, Söllradl and Hengge^[13] deduced NMR parameters for all possible chlorodisilanes. The data presented here agree quite well with theirs.

Infrared and Raman Vibrational Spectra

Infrared and Raman vibrational spectra were recorded for 1,2-dichlorodisilane and 1,1,2-trichlorodisilane. The spectra at ambient temperatures possess more lines than predicted by the selection rules based on symmetry arguments, a clear indication for rotational isomerism. The Raman spectra at low temperatures confirm this prediction, as the intensity ratio of a number of pairs of bands changes with temperature. Table 3 summarizes the data that have been obtained for the gases (IR) and the liquids (Raman).

Figure 3 illustrates the effect of temperature on the Ra-

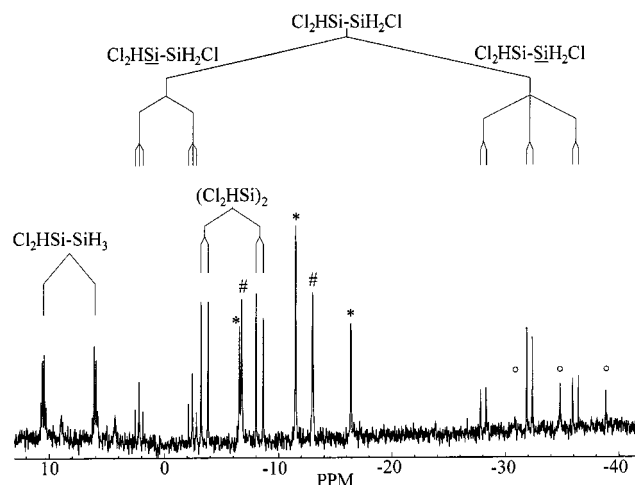


Figure 2. Proton coupled ^{29}Si NMR spectrum of 1,1,2-trichlorodisilane in the range $\delta = +10$ to -40 after equilibration with AlCl_3 ; lines marked with $\circ$, $*$ and $\#$ are due to H_3SiCl , H_2SiCl_2 and HSiCl_3 , respectively

Table 3. Absorption frequencies and assignments in the infrared spectra (gas, 2200–280 cm^{-1}) and Raman spectra (liquid, 2200–150 cm^{-1}) of 1,2-dichlorodisilane and 1,1,2-trichlorodisilane

$\text{ClH}_2\text{SiSiH}_2\text{Cl}$		$\text{ClH}_2\text{SiSiHCl}_2$		Assignment
IR (gas)	Raman (l)	IR (gas)	Raman (l)	
2193 s		2200 vs	2193 vs,sh	SiH stretch
2181 vs		2192 vs	2180 vs	SiH stretch
2170 s	2171 vs			SiH stretch
949 s				SiH_2 deformation
935 s	933 m	928 m	924 m,br	SiH_2 deformation
	921 m			SiH_2 deformation
	908 w			SiH_2 deformation
884 m		883 w		SiH_2 deformation
876 m	870 vw	876 s		SiH_2 deformation
843 vw	840 vw			ClSiH deformation
835 vw	832 vw	835 m	832 vw	ClSiH deformation
812 s		815 m	815 vw	ClSiH deformation
800 vs	782 vw	783 s	791 m	ClSiH deformation
766 s	757 vw		760 vw	SiH_2 twist
	702 m,b			SiH_2 twist
	691 w,sh	682 vw	688 m,br	SiH_2 twist
677 vw		621 m		SiH_2 twist
	567 vw, sh	582 vs	571 m,sh	SiCl stretch
540 m,br	540 m,br		550 s	SiCl stretch
	514 w,sh	528 s	525 w	SiCl stretch
			517 vw	SiH_2 rock
		495 vw	495 m	SiCl stretch
	441 s		424 m,sh	SiSi stretch
404 vw	411 m, sh	406 w	407 vs	SiSi stretch
	399 vs		396 vs, sh	SiSi stretch
358 w				SiH_2 rock
	308 vw			
			248 w	SiCl_2 deformation
			216 w,sh	SiCl_2 deformation
	195 vw		209 m	ClSiSi deformation
			180 m	SiCl_2 deformation
	157 m,br		151 s	ClSiSi deformation

man spectrum of 1,1,2-trichlorodisilane. For instance, the relative intensities within the line triple at 396/407/424 cm^{-1} , and of the line pairs at 209/216, 517/525 and 550/571

cm^{-1} are clearly temperature dependent. Similar effects are observed for 1,2-dichlorodisilane. As it is impossible to accurately assign the spectra of the conformers without the help of *ab initio* calculations of harmonic frequencies, only approximate descriptions of the modes are given in Table 3 without any reference to individual rotamers.

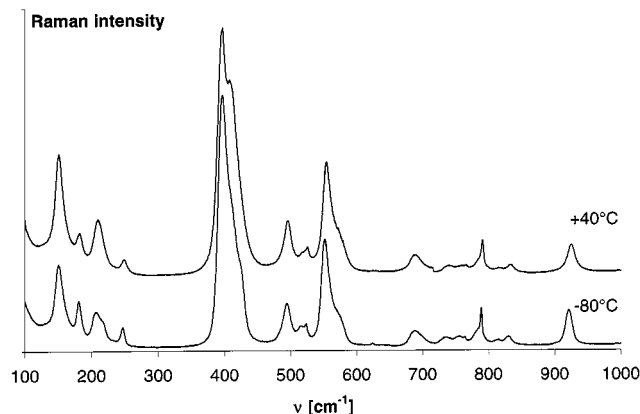


Figure 3. Raman spectra in the range $\tilde{\nu} = 100\text{--}1000\text{ cm}^{-1}$ of 1,1,2-trichlorodisilane at two different temperatures

So far, the rotational isomerism of 1,1,2,2-tetrachlorodisilane has been examined by electron diffraction^[33] and variable temperature Raman spectroscopy^[34] with the result that the *gauche* conformer is found to be more stable in both the gas and the liquid phase. *Ab initio* calculations at various levels of theory predict the *anti* rotamer as the low energy conformer. We will report on detailed investigations of the rotational isomerism of the title molecules, including spectra of the deuterated isotopomers as well as electron diffraction experiments, in a forthcoming publication.

Experimental Section

General: Most chlorinated organosilanes are sensitive towards moisture with the formation of Si–O–Si bonds and hydrogen chloride. Furthermore, hydriochlorosilanes are also sensitive towards oxygen, many of them igniting spontaneously on contact with air. All manipulations were therefore carried out under an atmosphere of dry, oxygen-free nitrogen. All solvents were dried by distillation from potassium prior to use.

Raman spectra were recorded with a T64000 spectrometer from Jobin-Yvon, equipped with a triple monochromator. The spectra were excited with the green line (514 nm) of an Ar laser. The chlorodisilanes were condensed into 1 mm capillary glass tubes which were sealed under an inert N_2 atmosphere.

The gas-phase infrared spectra of the hydriochlorodisilanes were recorded with a Perkin–Elmer 883 spectrometer using a Perkin–Elmer gas cell with a path length of 10 cm.

NMR spectra were recorded with a Bruker MSL 300 spectrometer as solutions in mesitylene (hydriochlorodisilanes) or toluene (naphthylidisilanes) using a capillary with D_2O as an external standard. Standard pulse sequences such as INEPT were employed.

Synthesis of 1,1,2-trichlorotrinaphthylidisilane: A solution of hexachlorodisilane (45.0 g, 167.4 mmol) in 100 mL of toluene was

added dropwise to a suspension of naphthyllithium in toluene [594.0 mmol; prepared from 1-bromonaphthalene (123.0 g, 594.0 mmol) and *n*-butyllithium]. A temperature of $-20\text{ }^\circ\text{C}$ was maintained throughout the procedure. Upon completion, the reaction mixture was refluxed for three hours. The salts were then separated by filtration and the solvent removed by evaporation in vacuo. The liquid residue was fractionated in vacuo. All volatile products up to a temperature of $120\text{ }^\circ\text{C}$ (0.01 Torr) were collected. They comprised a mixture of naphthyltrichlorosilane, pentachloronaphthylidisilane and tetrachlorodinaphthylidisilane. The residue that solidified at room temperature was found to be a mixture of 1,1,2-trichlorotrinaphthylidisilane and 1,2-dichlorotetranaphthylidisilane. Crystallization from toluene/petroleum ether (1:5) afforded 34.8 g (38%) of pure trichlorotrinaphthylidisilane. – $\text{C}_{30}\text{H}_{21}\text{Cl}_3\text{Si}_2$ (544.0): calcd. C 66.23, H 3.89; found C 66.45, H 3.86.

Synthesis of 1,1,2-Trinaphthylidisilane: The hydridodisilane was obtained in quantitative yields by standard reduction of the trichlorotrinaphthylidisilane with LiAlH_4 in diethyl ether. After addition of a solution of lithium aluminum hydride to the solution of the chlorodisilane at $0\text{ }^\circ\text{C}$, the reaction mixture was refluxed for several hours. Subsequently, it was added dropwise to $2\text{ N H}_2\text{SO}_4$ at a temperature of $0\text{ }^\circ\text{C}$. The organic layer was then separated and the diethyl ether removed in vacuo. The residue was recrystallized from petroleum ether giving the desired product as colorless crystals. – $\text{C}_{30}\text{H}_{24}\text{Si}_2$ (440.7): calcd. C 81.76, H 5.49; found C 81.75, H 5.59.

Synthesis of 1,2-Dichlorodisilane: 1,2-Dinaphthylidisilane^[31] (5.0 g) or the equivalent amount of 1,2-dimesityldisilane^[35] was placed in a thick-walled glass tube. A large excess of hydrogen chloride was then condensed into the tube, which was sealed carefully. The reaction mixture was cooled to $-70\text{ }^\circ\text{C}$ for five hours. The glass tube was then transferred to a vacuum line and the unreacted hydrogen chloride removed very carefully. 1,2-Dichlorodisilane was then separated from naphthalene (or mesitylene) by fractional condensation, as a colorless liquid. The yield was quantitative.

Synthesis of 1,1,2-Trichlorodisilane: In a procedure completely analogous to the synthesis of 1,2-dichlorodisilane, 1,1,2-trichlorodisilane was obtained in quantitative yields from 1,1,2-trinaphthylidisilane and liquid HCl. It was purified by fractional condensation.

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