

Long-chain alkanes with an internal $-H_2Si-SiH_2-$ linkage¹

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Abstract

1,2-Bis(*p*-tolyl)disilane (**2**) has been prepared through Wurtz-coupling of chloro(*p*-tolyl)silane using lithium metal. It was used as a precursor for 1,2-bis(trifluoromethanesulfonyloxy)disilane (**1**), which was obtained by treatment with two equivalents of trifluoromethanesulfonic acid. This intermediate was treated with *n*-BuLi, *n*-HexLi or *n*-OctMgBr to give the title compounds $(n-C_nH_{2n+1})SiH_2-SiH_2(n-C_nH_{2n+1})$ ($n = 4, 6, 8$), **4a–c**. Treatment of **2** with only one equivalent of triflic acid gave *p*-TolSiH₂SiH₂OSO₂CF₃ (**5**), which can be selectively converted into mixed alkyl-aryl-disilanes with LiR reagents, e.g. (*n*-Bu)SiH₂SiH₂(*p*-Tol), **6**. The crystal structure of compound **2** has been determined by an X-ray diffraction study; it has a staggered trans-conformation with a crystallographic center of inversion.

Keywords: Disilanes; Silaalkanes; Silanes; Si–Si coupling; Si–C cleavage; Arylsilanes

1. Introduction

There is considerable current interest in oligosilanes owing mainly to their unique photochemical and thermal properties, as well as to their ability to undergo catalytic dehydrogenative or desilanative coupling [1–3]. Si–Si bonds have an intense absorption (and luminescence) in the UV and near visible region [4] and can be subjected to a variety of photochemical transformations. Thermally or catalytically induced coupling reactions cause cross-linking of low molecular weight precursors to give new materials, the properties of which depend on the substitution pattern of the monomers.

In order to provide access to silicon-based materials with flexible, strongly hydrophobic side-chains, we became interested in long-chain hydrocarbons with internal, fully hydrogenated disilanediy units. In this report we describe a facile and versatile synthesis of such compounds, which are also expected to be interesting materials for surface coating through anchoring at the central disilane units [5].

Disilanes amenable to nucleophilic substitution in 1,2-positions are still rare. 1,2-Dichlorodisilane [6] is difficult to prepare in a pure state and cannot be stored for prolonged periods under standard conditions. Its

reactions are often accompanied by hydrogen shifts to give products with a modified substitution pattern. The analogous bromo- and iodo-disilanes are ill-characterized and offer no advantages [7]. Some of these preparative difficulties can be circumvented by the use of 1,2-bis(trifluoromethanesulfonyloxy)disilane [8] (**1**) first prepared recently from 1,2-diphenyldisilane and triflic acid in an inert solvent [8,9].

During our earlier investigations of the regioselectivity of Si–R bond cleavage using triflic acid [10], we observed that the experiments with *p*-tolyl and *p*-anisyl instead of phenyl groups always led to superior yields of pure products with high selectivity under less stringent conditions [11]. The present work was therefore undertaken based mainly on the *p*-tolyl-substituted precursor.

2. Results and discussion

Wurtz coupling of chloro(*p*-tolyl)silane [12] using lithium in hexane–diethyl ether affords 1,2-bis(*p*-tolyl)disilane (**2**) [13] in 55% yield (m.p. 36–37°C, b.p. 114°C/0.05 Torr). The structure of the product has been determined in an X-ray diffraction study. The molecule has a staggered trans-conformation with a crystallographically imposed center of inversion, as shown in Fig. 1. A complete set of analytical and spectroscopic data is also available (Section 3).

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¹ Dedicated to Professor Robert Corriu in recognition of his contributions to organosilicon chemistry.

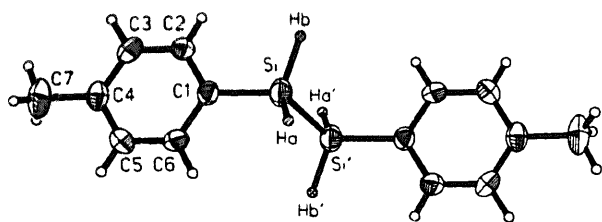


Fig. 1. Molecular structure of (*p*-Tol)SiH₂–SiH₂(*p*-Tol) with atomic numbering (ORTEP, 50% probability ellipsoids). Selected bond lengths (Å) and angles (deg) are as follows: Si–Si', 2.329(2); Si–Cl, 1.863(2); Si–Ha, 1.42(2); Si–Hb, 1.45(3); Ha–Si–Cl, 108.5(9); Hb–Si–Cl, 108(1); Ha–Si–Hb, 114(1); Si'–Si–Ha, 105.9(9); Si'–Si–Hb, 107(1).

The analogous Wurtz-coupling with *p*-anisyl(chloro)silane [14] is less satisfactory. Under the conditions optimized for **2** above, bis(*p*-anisyl)silane is the main product. The coupling reaction does take place in hexane-free diethyl ether to give low yields of 1,2-bis(*p*-anisyl)disilane (**3**), but not in excess of 20%. Moreover, all attempts to purify compound **3** by distillation or crystallization met with failure owing to extensive decomposition. Again disproportionation to give (*p*-MeOC₆H₄)SiH₃ and (*p*-MeOC₆H₄)₂SiH₂ is the dominant decomposition pathway. Compound **3** and the decomposition products have been characterized spectroscopically.

All further studies were therefore conducted exclusively with compound **2**, which is an excellent starting material for a convenient preparation of the 1,2-bis(triflate)disilane (**1**) using two equivalents of trifluoromethanesulfonic acid in toluene in the temperature range from –25 to –10°C (Scheme 1). The product is readily identified from its NMR spectra, preferentially by use of the diagnostic ²⁹Si NMR chemical shift (δ = 28 ppm) [8]. Solutions of **1** in toluene–hexane react with two equivalents of *n*-alkyllithium or *n*-alkylmagnesiumbromide compounds at –10 to –20°C to afford high yields of the corresponding 1,2-bis(alkyl)disilanes (**4a–c**). The products can be isolated as colorless liquids by fractional distillation under reduced pressure. Sam-

Table 1
Boiling points of the 1,2-dialkyl-disilanes **4a–c** and their all-carbon analogues

Compound	Boiling point (°C/Torr)	
<i>n</i> -Decane	63/15	173/760
<i>n</i> -Tetradecane	129.5/15	252.5/760
<i>n</i> -Octadecane	181.5/15	317/760
5,6-Disiladecane 4a	62/3	210/760 ^a
7,8-Disilatetradecane 4b [5]	70/0.05	290/760 ^a
9,10-Disilaoctadecane 4c	110/0.05	350/760 ^a

^a Extrapolated.

ples may be handled in air at ambient temperature for a short time without significant decomposition through oxidation or hydrolysis.

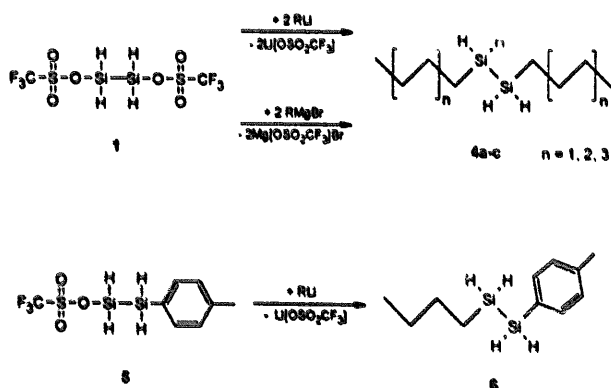
The composition was confirmed by elemental analyses and mass spectrometry data. The NMR spectra are fully consistent with symmetrical structures having equivalent halves (–SiH₂R). Compounds **4** are thus unbranched, long-chain disila-alkanes with a central disilane unit. Their physical properties are similar to those of the corresponding all-carbon analogues (*n*-decane, *n*-tetradecane, *n*-octadecane, Table 1). It should be noted that in the reaction mixture there is no evidence for partial rearrangement of the linear disila-alkanes to give 1,1-dialkyl-disilanes of the type H₃Si–SiHR₂. This is in contrast to reactions of **1** with alcohols or amines, which were found to give products with a redistribution of the Si-bonded hydrogen atoms, i.e. H₃Si–SiH(OR)₂ or H₃SiSiH(NR₂)₂ etc. [15]. It is only with amines bearing bulky groups R that 1,2-substitution predominates over 1,1-substitution of the disilane **1** [15].

Treatment of compound **2** with only one equivalent of triflic acid in toluene affords solutions of the 1-(*p*-tolyl)-2-(trifluoromethanesulfonyloxy)disilane **5** (Scheme 1). This triflate can be treated with one equivalent of *n*-butyllithium to give 1-(*n*-butyl)-2-(*p*-tolyl)disilane **6** in 60% yield as a colorless, distillable liquid (b.p. 77°C/0.1 Torr), which is also readily identified from its analytical and spectroscopic data. Again the 1,2-substitution is the sole ligand pattern in the product, and no scrambling of alkyl or aryl groups occurs. This result shows that alkylations of **1** are fully regioselective.

3. Experimental

3.1. General

All experiments were routinely carried out under pure dry nitrogen. Solvents were dried, degassed and saturated with nitrogen, glassware was oven-dried and filled with nitrogen. Standard analytical and spectro-



Scheme 1.

sopic equipment was used throughout. Reagents were prepared by published procedures [12–14].

3.2. Preparation of 1,2-bis(*p*-tolyl)disilane (2)

A solution of chloro(*p*-tolyl)silane (90.0 g, 0.58 mol) in a mixture of hexane (400 ml) and diethyl ether (100 ml) is stirred with lithium powder (6.0 g, 0.87 mol) for 36 h at 20°C. The mixture is filtered and the filtrate distilled in a vacuum. The colorless liquid product **2** (yield 38.7 g, 55%) is collected at boiling point (b.p.) 114°C/0.05 Torr. It crystallizes in the receiver, melting point (m.p.) 36–37°C. MS (EI, 70 eV, GC-coupled): $m/z = 242$ (M^+). Raman (crystals): 2111.0 cm^{-1} (νSiH), 446.9 cm^{-1} (νSiSi).

^1H NMR (C_6D_6 , 25°C): δ 2.05 (s, 6H, Me), 4.55 (s, $J(\text{Si,H}) = 191.9$ Hz, 4H, SiH_2), 6.92 (d, $J(\text{H,H}) = 7.9$ Hz, 4H, $\text{H}_{3,5}$), 7.41 (d, $J(\text{H,H}) = 7.9$ Hz, 4H, $\text{H}_{2,6}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (v.s.): δ 21.4 (s, Me), 125.5 (s, C_1), 129.4 (s, $\text{C}_{3,5}$), 136.4 (s, $\text{C}_{2,6}$), 139.5 (s, C_4). ^{29}Si NMR (DEPT, v.s.): δ -61.8 (ttt, $J(\text{Si,H}) = 191.9$ Hz, $J(\text{H,H}) = 6.4$ and 6.4 Hz respectively).

3.3. Preparation of 1,2-bis(*p*-anisyl)disilane (3)

A solution in diethyl ether (100 ml) of chloro(*p*-anisyl)silane, prepared in situ from *p*-anisylsilane (10.0 g, 72 mmol) and BCl_3 (2.8 g, 24 mmol), is stirred and treated with lithium powder (0.60 g, 87 mmol) for 48 h with stirring. The mixture is filtered and the filtrate fractionally distilled under vacuum until the flask temperature reaches 60°C. The main volatile component is *p*-anisylsilane. The residue consists of a mixture of **3** (70%) and bis(*p*-anisyl)silane (20%) with other impurities, as derived from GLC-MS analysis. **3**: MS (EI, 70 eV) $m/z = 274$ (M^+). ^1H NMR (v.s.): δ 3.29 (s, 6H, OMe), 4.47 (s, 4H, SiH_2), 6.72 (d, $J(\text{H,H}) = 8.5$ Hz, 4H, $\text{H}_{3,5}$), 7.49 (d, $J(\text{H,H}) = 8.5$ Hz, 4H, $\text{H}_{2,6}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (v.s.): δ 54.6 (s, OMe), 114.4 (s, $\text{C}_{3,5}$), 119.2 (s, C_1), 137.8 (s, $\text{C}_{2,6}$), 161.6 (s, C_4). ^{29}Si NMR (DEPT, v.s.): δ -62.1 (ttt, $J(\text{Si,H}) = 191.4$ Hz, $J(\text{H,H}) = 6.4$ and 6.4 Hz).

3.4. Preparation of 1,2-bis(trifluoromethanesulfonyl)disilane (1)

A solution of compound **2** (4.0 g, 16.5 mmol) in toluene (100 ml) is cooled to -25°C with stirring and treated with triflic acid (4.95 g, 33.0 mmol). Stirring is continued for 1 h at -10°C. The resulting solution is used in all further experiments. ^1H NMR (d_8 -toluene, -15°C): δ 4.47 (s, $J(\text{Si,H}) = 272$ Hz, 4H, SiH_2). ^{29}Si NMR (DEPT, v.s.): δ -28.2 (tm, $J(\text{Si,H}) = 272$ Hz).

3.5. Preparation of 1,2-bis(*n*-butyl)disilane (4a)

A freshly prepared solution of **1** (from 4.0 g (16.5 mmol) of **2**) in toluene (100 ml) is treated slowly with a solution of *n*-butyllithium (2.1 g, 33.0 mmol) in hexane (25 ml) at -10°C with stirring. The mixture is then allowed to warm to 20°C. After completion of the reaction (ca. 1–2 h) the lithium triflate precipitate is filtered off and the filtrate fractionally distilled under vacuum. Colorless liquid (yield 2.0 g, 71%), b.p. 62°C/3 Torr. MS (EI, 70 eV): $m/z = 174$ (M^+). ^1H NMR (C_6D_6 , 25°C): δ 0.68 (m, 4H, CH_2Si), 0.80 (t, $J(\text{H,H}) = 7.3$ Hz, 6H, Me), 1.25 (m, 4H, CH_2CSi), 1.33 (sex, $J(\text{H,H}) = 7.3$ Hz, 4H, CH_2Me), 3.69 (s, $J(\text{Si,H}) = 183$ Hz, 4H, SiH_2). $^{13}\text{C}\{^1\text{H}\}$ NMR (v.s.): δ 7.3 (s, CH_2Si), 13.9 (s, Me), 26.2 (s, CH_2CSi), 30.0 (s, CH_2Me). ^{29}Si NMR (DEPT, v.s.): δ -63.6 (tm, $J(\text{Si,H}) = 183$ Hz). Anal. Found: C, 55.38; H, 12.59. $\text{C}_8\text{H}_{22}\text{Si}_2$ (174.44 g mol^{-1}). Calc.: C, 55.08; H, 12.71%.

3.6. Preparation of 1,2-bis(*n*-hexyl)disilane (4b) [5]

As described for **4a**, a solution of **1** (from 1.0 g (4.1 mmol) of **2**) in toluene (25 ml) is treated with a solution of *n*-hexyllithium (0.76 g, 8.2 mmol) in hexane (3.3 ml) at -20°C. The mixture is filtered and the filtrate fractionally distilled under vacuum to give a colorless liquid (yield 0.65 g, 68%), b.p. 70°C/0.05 Torr. MS (EI, 70 eV): $m/z = 230$ (M^+). ^1H NMR (C_6D_6 , 25°C): δ 0.74 (m, 4H, CH_2Si), 0.87 (t, $J(\text{H,H}) = 7.3$ Hz, 6H, Me), 1.2–1.3 (m, 12H, CH_2), 1.39 (sex, $J(\text{H,H}) = 7.3$ Hz, 4H, CH_2Me), 3.74 (s, $J(\text{Si,H}) = 183$ Hz, 4H, SiH_2). $^{13}\text{C}\{^1\text{H}\}$ NMR (v.s.): δ 7.6 (s, CH_2Si), 14.3 (s, Me), 23.0 (s, CH_2), 27.8 (s, CH_2), 31.8 (s, CH_2), 32.8 (s, CH_2). ^{29}Si NMR (DEPT, v.s.): δ -63.5 (tm, $J(\text{Si,H}) = 183$ Hz). Anal. Found: C, 62.82; H, 13.09. $\text{C}_{12}\text{H}_{30}\text{Si}_2$ (230.54 g mol^{-1}). Calc.: C, 62.52; H, 13.12%.

3.7. Preparation of 1,2-bis(*n*-octyl)disilane (4c)

A freshly prepared solution of **1** (from 1.0 g (4.1 mmol) of **2**) in toluene (25 ml) is treated slowly with a solution of *n*-octylmagnesium bromide (1.79 g, 8.2 mmol) in tetrahydrofuran (4.2 ml) at -20°C with stirring. After being allowed to warm to 20°C the mixture is filtered and the volume of the solvent reduced to one-half. Addition of hexane leads to precipitation of the remaining magnesium salts. After a second filtration the filtrate is fractionally distilled under vacuum to give a colorless liquid (yield 0.8 g, 68%), b.p. 110°C/0.05 Torr. MS (EI, 70 eV): $m/z = 286$ (M^+). ^1H NMR (C_6D_6 , 25°C): δ 0.75 (m, 4H, CH_2Si), 0.89 (t, $J(\text{H,H}) = 7.3$ Hz, 6H, Me), 1.2–1.3 (m, 20H, CH_2), 1.41 (sex, $J(\text{H,H}) = 7.3$ Hz, 4H, CH_2Me), 3.72 (s,

$J(\text{Si,H}) = 183 \text{ Hz}$, 4H, SiH_2). $^{13}\text{C}\{^1\text{H}\}$ NMR (v.s.): δ 7.7 (s, CH_2Si), 14.4 (s, Me), 23.1 (s, CH_2), 27.9 (s, CH_2), 29.7 (s, $2\times\text{CH}_2$), 32.4 (s, CH_2), 33.2 (s, CH_2). ^{29}Si NMR (DEPT, v.s.): δ -63.5 (tm, $J(\text{Si,H}) = 183 \text{ Hz}$). Anal. Found: C, 67.21; H, 13.51. $\text{C}_{16}\text{H}_{38}\text{Si}_2$ (286.65 g mol^{-1}). Calc.: C, 67.04; H, 13.36%.

3.8. Preparation of 1-(*p*-tolyl)-2-(trifluoromethanesulfonyloxy)disilane (5)

A solution of compound 2 (5.0 g, 20.6 mmol) in toluene (150 ml) is cooled to -25°C and treated slowly with triflic acid (3.1 g, 20.6 mmol) with vigorous stirring. After stirring for 1 h at -10°C the mixture is used for further reactions.

3.9. Preparation of 1-(*n*-butyl)-2-(*p*-tolyl)disilane (6)

A freshly prepared solution of 5 (from 5.0 g (20.6 mmol) of 2) in toluene (150 ml) is treated slowly with a solution of *n*-butyllithium in hexane (1.32 g, 20.6 mmol) at -10°C with stirring. The mixture is warmed to 20°C and filtered, and the filtrate is fractionally distilled under vacuum to give a colorless liquid (yield 2.64 g, 61.5%), b.p. $77^\circ\text{C}/0.1 \text{ Torr}$. MS (EI, 70 eV): $m/z = 208$ (M^+). ^1H NMR (C_6D_6 , 25°C): δ 0.69 (m, 2H, CH_2Si), 0.75 (t, $J(\text{H,H}) = 7.3 \text{ Hz}$, 3H, MeCH_2), 1.20 (qui, $J(\text{H,H}) = 7.3 \text{ Hz}$, 2H, CH_2CSi), 1.29 (sex, $J(\text{H,H}) = 7.3 \text{ Hz}$, 2H, CH_2Me), 2.06 (s, 3H, MeC_4), 3.76 (m, $J(\text{Si,H}) = 185 \text{ Hz}$, 2H, SiH_2CH_2), 4.39 (t, $J(\text{H,H}) = 2.4 \text{ Hz}$, $J(\text{Si,H}) = 192 \text{ Hz}$, 2H, SiH_2C_1), 6.92 (d, $J(\text{H,H}) = 7.6 \text{ Hz}$, 2H, $\text{H}_{3/5}$), 7.39 (d, $J(\text{H,H}) = 7.6 \text{ Hz}$, 2H, $\text{H}_{2/6}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (v.s.): δ 7.1 (s, CH_2Si), 14.0 (s, MeCH_2), 21.6 (s, MeC_4), 26.2 (s, CH_2CSi), 29.9 (s, CH_2Me), 126.3 (s, C_1), 129.5 (s, $\text{C}_{3/5}$), 136.4 (s, $\text{C}_{2/6}$), 139.4 (s, C_4). ^{29}Si NMR (DEPT, v.s.): δ -62.5 (tm, $J(\text{Si,H}) = 185 \text{ Hz}$, SiH_2CH_2), -62.4 (tm, $J(\text{Si,H}) = 192 \text{ Hz}$, SiH_2C_1). Anal. Found: C, 63.54; H, 9.84. $\text{C}_{11}\text{H}_{20}\text{Si}_2$ (208.45 g mol^{-1}). Calc.: C, 63.38; H, 9.67%.

4. Crystal structure determination

The sample was mounted in a glass capillary on an Enraf–Nonius CAD4 diffractometer and used for measurements of precise cell constants and intensity data collection. During data collection, three standard reflections were measured periodically as a general check of crystal and instrument stability. No significant changes were observed. Graphite-monochromated $\text{Mo K}\alpha$ radiation was used. The structure was solved by direct methods and refined by full matrix least squares calculations on F (SHELXTL-PC).

Crystal data: $\text{C}_{14}\text{H}_{18}\text{Si}_2$, $M_r = 242.47$, colorless crystals of dimensions $0.2 \times 0.3 \times 0.8 \text{ mm}^3$, mono-

clinic, $a = 6.353(1)$, $b = 7.434(1)$, $c = 15.263(2) \text{ \AA}$, $\beta = 92.00(1)^\circ$, space group $P2_1/c$, $Z = 2$, $V = 711.6(1) \text{ \AA}^3$, $\rho_{\text{calc}} = 1.132 \text{ g cm}^{-3}$, $F(000) = 260$; $T = -62^\circ\text{C}$. Data were corrected for Lorentz and polarization effects but not for absorption ($\mu(\text{Mo K}\alpha) = 2.2 \text{ cm}^{-1}$). 1264 measured ($(\sin \theta / \lambda)_{\text{max}} = 0.62 \text{ \AA}^{-1}$) reflections, 945 observed reflections ($F_o \geq 4\sigma(F_o)$) were used for refinement. All hydrogen atoms were found and refined isotropically, all non-H atoms were refined with anisotropic displacement parameters. The structure converged for 109 refined parameters to an R (R_w) value of 0.0357 (0.0357). Residual electron densities: $+0.26 / -0.24 \text{ e \AA}^{-3}$. Selected bond lengths and angles are given in the corresponding figure caption, more information on the crystal structure determination can be obtained from the Fachinformationszentrum Karlsruhe GmbH, D-76344 Eggenstein-Leopoldshafen on quoting the names of the authors, the journal citation and the CSD number 405442.

Acknowledgements

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