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REACTIONS OF TRIS(TRIMETHYLSILYL)METHYLSILICON COMPOUNDS WITH ORGANOLITHIUM REAGENTS *

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Summary

The compounds $TsiSiH_3$, $TsiSiH_2F$, and $TsiSiH_2I$ ($Tsi = (Me_3Si)_3C$) have been shown to react with p-YC_6H_4Li ($Y = H$, Me, MeO, Cl, or CF_3) compounds in ether/hexane to give the corresponding compounds $TsiSiH_2(C_6H_4Y)$; use of $TsiSiH_2F$ gives the best results. When the p-YC_6H_4Li is made from p-YC_6H_4Br and BuLi appreciable amounts of $TsiSiH_2Bu$ can appear among the products, and $TsiSiH_3$ has been shown to react with BuLi to give $TsiSiH_2Bu$, which on treatment with more BuLi gives $TsiSiHBu_2$. Reaction of PhLi with TsiSiHBuF gives a high yield of TsiSiHBuPh. Such reactions provide a new route to a range of compounds containing the Tsi group attached to a functional silicon centre.

Treatment of the compounds $TsiSiH_2(C_6H_4Y\text{-}p)$ with I_2 in CCl_4 has given the corresponding iodides $TsiSiH(C_6H_4Y\text{-}p)I$.

Introduction

The mechanisms of reactions of compounds of the types $TsiSiR_2I$ ($Tsi = (Me_3Si)_3C$) and $(Me_3Si)_2C(SiMe_2Z)SiR_2X$ (e.g., $Z = OMe$ or $CH{=}CH_2$) [1] are of considerable interest, and to throw further light on those mechanisms we wished to study the effects of substituents Y in compounds of the type $TsiSiH(C_6H_4Y\text{-}p)I$. The need to make such compounds presented an opportunity to examine the possibility of attaching the C_6H_4Y-p ligand to a silicon centre already bearing a Tsi group, instead of using the usual approach of treating TsiLi with, e.g., $YC_6H_4SiCl_3$ (cf. ref. 2). It was previously shown that treatment of $TsiSiCl_3$ with PhLi did not result in introduction of Ph in place of Cl [2], but more recently Fritz and his

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* Dedicated to Professor L. Sacconi in recognition of his important contribution to organometallic chemistry.

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colleagues reported the formation of some $TsiSiMe_2CH_2SiMe_2Ph$ on prolonged treatment of $TsiSiMe_2Cl$ with $LiCH_2SiMe_2Ph$ in the presence of TMEDA in refluxing THF [3]. We thus decided to examine the reactions of aryllithium reagents with the compounds $TsiSiH_2X$ (X = H, F, or I), since it is known that the presence of two hydrogen atoms, or even one such atom, on a silicon centre bearing a Tsi group greatly increases the ease of nucleophilic substitution at that centre [2]. When the present study was complete, a report by Sakurai and his colleagues revealed that $TsiSiF_3$ reacts with mesityllithium to give $TsiSiF_2(C_6H_2Me_3\text{-}2,4,6)$ [4].

Results and discussion

We initially examined the reaction of $TsiSiH_3$ with a 1.9 molar proportion of $p\text{-}MeC_6H_4Li$ formed by treatment of $p\text{-}MeC_6H_4Br$ in ether with BuLi * in hexane. After 4 h under reflux, work-up (beginning with addition of aqueous NH_4Cl) followed by GLC analysis of the product mixture, showed that very little $TsiSiH_3$ remained, and that $TsiSiH_2C_6H_4Me\text{-}p$ constituted ca. 50% and $TsiSiH_2Bu$ 25% of the mixture, the remainder being made up of several minor by-products which were not investigated further: the $TsiSiH_2(C_6H_4Me\text{-}p)$ was isolated by sublimation. The corresponding procedure starting from $p\text{-}ClC_6H_4Br$ (but with a shorter reflux) gave a mixture of $TsiSiH_2C_6H_4Cl\text{-}p$, $TsiSiH_2Bu$, and $TsiSiH_3$ in ca. 85/14/1 ratio. With $p\text{-}CF_3C_6H_4Br$ as starting material (but with some THF in the solvent mixture) $TsiSiH_2(C_6H_4CF_3\text{-}p)$ formed ca. 33% of the isolated mixture (see Table 1), but when $p\text{-}MeOC_6H_4Br$ was used only a trace of $TsiSiH_2C_6H_4OMe\text{-}p$ was formed, along with $TsiSiH_2Bu$ (50%) and unchanged $TsiSiH_3$ (50%).

In view of the results of reactions with $TsiSiH_2I$ and $TsiSiH_2F$, described later, it seems unlikely that the formation of so much $TsiSiH_2Bu$ in the above reactions was the result of inadequate reaction of the bromides $p\text{-}YC_6H_4Br$ with the BuLi before addition of $TsiSiH_3$. Presumably there is an equilibrium $BuLi + p\text{-}YC_6H_4Br \rightleftharpoons BuBr + p\text{-}YC_6H_4Li$, which normally lies well over to the right, but the BuLi reacts markedly more readily than the aryllithium compound with the $TsiSiH_3$. It would evidently be better in the reactions with $TsiSiH_3$ to use XC_6H_4Li reagents made from XC_6H_4Br and metallic Li.

Better results were obtained when $TsiSiH_2I$ or $TsiSiH_2F$ was used in place of $TsiSiH_3$. Thus reaction of $TsiSiH_2I$ with $p\text{-}MeOC_6H_4Li$ made from the bromide and BuLi gave a product mixture which was shown by GLC to contain $TsiSiH_2C_6H_4OMe\text{-}p$ (92%), $TsiSiH_2Bu$ (4%), and $TsiSiH_3$ (1%), along with minor amounts of other species, and the $TsiSiH_2C_6H_4OMe\text{-}p$ was isolated in good yield. When $p\text{-}MeOC_6H_4Li$ made from metallic Li was used, $TsiSiH_2C_6H_4OMe\text{-}p$ constituted ca. 90% of the product mixture, and $TsiSiH_2Bu$ was, of course, absent. Reactions with $p\text{-}MeC_6H_4Li$ and $p\text{-}CF_3C_6H_4Li$ (both made as usual from the bromide and BuLi) gave 90 and 60% yields, respectively, of the expected products (see Table 1).

Even better results were obtained when the fluoride $TsiSiH_2F$ was used as the starting material. The $TsiSiH_2C_6H_4Y\text{-}p$ compounds were formed in yields of ca. 90% or more in all cases (see Table 1), even though the reaction with $p\text{-}MeC_6H_4Li$ was carried out at room temperature and for only 2 h.

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* Bu denotes n-Bu throughout.

TABLE 1

DETAILS OF REACTIONS OF $TsiSiH_2X$ SPECIES WITH p-YC_6H_4Li [a]

X (mmol) [b]	Y (mmol [c]	Solvent (cm^3) [E = Et_2O; H = C_6H_{14}]	Time (h)	Product composition (%) [d] [Ar = $TsiSiH_2C_6H_4Y$; Bu = $TsiSiH_2Bu$; SM = $TsiSiH_2X$]
$TsiSiH_3$ (1.0)	Me(1.9)	E(10)/H(0.8)	4	Ar, 50; Bu, 25; others 25
(4.0)	MeO(4.0)	E(10)/H(1.7)/ T(10) [e]	1	Bu, 50; SM(50)
(1.0)	Cl(1.3)	E(8)/H(1.3)	3	Ar, 85; Bu, 14; SM(1)
(2.0)	CF_3(3.0)	E(10)/H(1.2)	5	Ar, 40; Bu, 5; SM, 5; others (50)
$TsiSiH_2I$(1.0)	Me(1.44)	E(10)/H(0.6)	2	Ar, 90; Bu, 1; others, 9
	MeO(1.44)	E(8)/H(0.5)	5	Ar, 92; Bu, 4: SM, 1: others, 3
	MeO(1.44) [f]	E(10) [f]	3	Ar, 90; others 10
	CF_3(1.44)	E(8)/H(0.6)	2.5	Ar, 60; Bu, 15; others, 25.
$TsiSiH_2F$(1.0)	Me(1.2)	E(10)/H(0.5)	2 [g]	Ar, 90; Bu, 1; others, 9.
	MeO(1.2)	E(10)/H(0.5)	2 [g]	Ar, 90; SM, 8; others, 2.
	Cl(1.1)	E(7)/H(0.50)	3	Ar, 90; SM, 8; others 2
	CF_3(1.1)	E(7)/H(0.5)	2	Ar, 98; Bu, 1; SM, 1.

[a] The p-YC_6H_4Li was made from p-YC_6H_4Br and n-BuLi in hexane (see Experimental section) unless otherwise indicated; reaction times refer to the period under reflux unless otherwise indicated. [b] (mmol) denotes amount of $TsiSiH_2X$ used. [c] (mmol) denotes amount of p-YC_6H_4Br initially taken. [d] Approximate composition as derived from analysis by GLC/MS. [e] T = THF. [f] The YC_6H_4Li was made from p-YC_6H_4Br and Li. [g] At room temperature.

As expected in the light of results described above, $TsiSiH_2Bu$ was formed almost quantitatively when $TsiSiH_3$ was treated with BuLi alone. More surprisingly, $TsiSiH_2Bu$ was itself found to react with BuLi to give a fairly good yield of $TsiSiHBu_2$. However, when $TsiSiH_2Bu$ was treated with a 1.5 molar proportion of PhLi at room temperature, a mixture of unchanged $TsiSiH_2Bu$ (ca. 40%) and Ph_3SiBu (ca 60%) was obtained; the latter was isolated and its identity confirmed. Apparently, when the PhLi attacks the $TsiSiH_2Bu$, possibly to give the 5-coordinate species $[TsiSiH_2BuPh]^-$ (which would be more sterically strained than $[TsiSiH_2Bu_2]^-$, the anion Tsi^- (which is a relatively stable carbanion [5]) leaves more readily than H^-, thereby relieving the steric strain, to give SiH_2PhBu, which then reacts with PhLi to give Ph_3SiBu. (The TsiH expected to be formed from TsiLi hydrolysis was presumably evaporated off during the removal of solvent from the product under reduced pressure in the final stages of the work-up.) In contrast, when TsiSiHBuF was used in the reaction with PhLi, the product mixture consisted of TsiSiHBuPh (90%) and TsiSiHFBu (10%), and the TsiSiHBuPh was isolated in 60% yield.

The dihydrides $TsiSiH_2(C_6H_4Y$-$p)$ were in each case converted into the required iodides $TsiSiH(C_6H_4Y$-$p)I$ by treatment with a molar proportion of ICl in CCl_4.

The reaction of $TsiSiH_2X$ species described above can clearly be of considerable value in synthesis of new highly sterically hindered organosilicon compounds. Yields of isolated products were low because of mechanical losses in the small-scale experiments, and there is no reason to doubt that they could be much improved by working on a larger scale.

Experimental

Materials and procedures

The compounds $TsiSiH_3$ [2] and $TsiSiH_2I$ [6] were made as previously described. Butyllithium was purchased from Aldrich as a 2.4 *M* solution in hexane, and the concentration checked before use.

Solvents were dried by standard methods.

All reactions involving organolithium reagents were carried out under dry N_2.

Spectra and GLC

The ^1H and ^{19}F NMR spectra were recorded (at 80 and 75.38 MHz, respectively) on a Bruker WP80 spectrometer and the ^{29}Si spectra (at 71.55 MHz, with gated decoupling and a 15 s relaxation delay) on a Bruker WM360 spectrometer. Chemical shifts were determined relative to internal $CHCl_3$ (δ 7.24) for ^1H, external $CFCl_3$ for ^{19}F, and external $SiMe_4$ for ^{29}Si.

Mass spectra were determined by electron impact at 70 eV. The organosilicon compounds identified by GLC/MS all showed strong $[M-\mathrm{Me}]^+$ peaks.

A column of 3% OV-17 on Gas Chrome Q was used for the linked GLC/MS analyses. The approximate percentages compositions of product mixtures are based on relative areas of peaks from a flame ionization detector.

1. Reactions of $TsiSiH_3$

(a) For the reaction with p-MeC_6H_4Li, a 2.4 *M* solution of BuLi in hexane (0.80 cm^3, 1.92 mmol of BuLi) was added dropwise with stirring to a solution of p-MeC_6H_4Br (0.33 g, 1.92 mmol) in ether (5 cm^3), and stirring was continued for 30 min at room temperature. A solution of $TsiSiH_3$ (0.26 g, 1.0 mmol) in ether (5 cm^3) was added, and the mixture was refluxed for 4 h then allowed to cool. Aqueous NH_4Cl was cautiously added with stirring, followed by additional ether, and the organic layer was separated, washed, and dried ($MgSO_4$). The solvent was taken off in a rotary evaporator, and the residue was kept under vacuum to leave a white solid. Analysis (of a solution in CCl_4) of this solid by GLC/MS revealed that it was made up of approximately 50% of $TsiSiH_2C_6H_4Me$-p, 15% of $TsiSiH_2Bu$, 1% of TsiH, and a total of 24% of several additional minor products. The major component was sublimed out under vacuum and shown to be $TsiSiH_2(C_6H_4Me$-$p)$, which was isolated in ca. 30% yield based on the aryl bromide initially used. The ^1H NMR data are given in Table 2.(b) Similar procedures starting from p-YC_6H_4Br with Y = MeO, Cl, and CF_3 (for details of amounts, reaction times etc., see Table 1) gave the product mixtures shown in Table 1. Some NMR data for isolated products are shown in Table 2. For the reaction starting from p-$CF_3C_6H_4Br$, stirring was continued for 1 h after addition of BuLi, until a deep orange solution was formed. The residue after work-up was an orange oil (which was analysed by GLC/MS), but washing of this with MeOH gave a solid, which was sublimed to give (white) $TsiSiH_2C_6H_4CF_3$-p in ca. 30% yield (Found: C, 50.2; H, 8.1. $C_{17}H_{33}F_3Si_4$ calc: C, 50.1; H, 8.1%).

(c) A solution of $TsiSiH_3$ (1.0 mmol) in ether (5 cm^3) was added to BuLi (1.0 mmol)

TABLE 2

NMR DATA

Compound	Chemical shifts (in ppm) and coupling constants [a]
TsiSiHPhI	δ(H) 0.32 (s, $SiMe_3$), 5.13 (s, SiH), 7.24–7.84 (m, Ar); δ(Si) 0.47 (s, $SiMe_3$), −24.68 (d, ^1J(SiH) 228.5 Hz, ^3J(Si–H) (to *o*-H) 6.4 Hz, SiHArI).
TsiSiH(C_6H_4Me-*p*)I	δ(H) 0.32 (s, $SiMe_3$), 2.35 (s, *p*-Me), 5.11 (s, SiH), 7.1–7.7 (m, Ar); δ(Si) 0.41 (s, $SiMe_3$), −24.72 (d, ^1J(SiH) 227.4 Hz, SiHArI).
TsiSiH(C_6H_4OMe-*p*)I	δ(H) 0.31 (s, $SiMe_3$), 3.80 (s, OMe), 5.10 (s, SiH), 6.80–7.75 (m, Ar); δ(Si) 0.35 (s, $SiMe_3$), −25.61 (d, ^1J(SiH) 227.4 Hz, SiHArI).
TsiSiH(C_6H_4Cl-*p*)I	δ(H) 0.31 (s, $SiMe_3$), 5.09 (s, SiH), 7.24–7.76 (m, Ar); δ(Si) 0.16 (s, $SiMe_3$), −26.25 (d, ^1J(SiH) 229.7 Hz, SiHArI).
TsiSiH($C_6H_4CF_3$-*p*)I	δ(H) 0.32 (s, $SiMe_3$), 5.13 (s, SiH), 7.52–7.96 (m, Ar); δ(F) −63.4; δ(Si) 0.22 (s, $SiMe_3$), −26.56 (d, ^1J(SiH) 230.8 Hz, SiHArI).
TsiSiHBuI	δ(H) 0.29 (s, $SiMe_3$), 0.79–1.58 (m, Bu), 4.59–4.66 (m, SiH) δ(Si) −0.38 (s, $SiMe_3$), −10.56 (d, ^1J(SiH) 221.1 Hz).
$TsiSiH_2Ph$	δ(H) 0.21 (s, $SiMe_3$), 4.56 (s, SiH), 7.24–7.81 (m, Ar).
$TsiSiH_2C_6H_4$Me-*p*	δ(H) 0.21 (s, $SiMe_3$), 2.34 (s, *p*-Me) 4.55 (s, SiH), 7.08–7.69 (m, Ar); δ(Si) −0.10 (s, $SiMe_3$), −31.36 (t, SiH_2, ^1J(SiH) 195.8 Hz).
$TsiSiH_2C_6H_4$OMe-*p*	δ(H) 0.20 (s, $SiMe_3$), 3.80 (s, OMe), 4.54 (s, SiH), 6.80–7.72 (m, Ar); δ(Si) −0.20 ($SiMe_3$), −31.81 (t, SiH_2, ^1J(SiH) 195.6, ^3J(SiH) (to *o*-H) 5.8 Hz).
$TsiSiH_2C_6H_4$Cl-*p*	δ(H) 0.19 (s, $SiMe_3$), 4.52 (s, SiH), 7.22–7.72 (m, Ar); δ(Si) −0.04 ($SiMe_3$), −31.12 (t, SiH_2, ^1J(SiH) 198.0, ^3J(SiH) (to *o*-H) 5.5 Hz).
$TsiSiH_2C_6H_4CF_3$-*p*	δ(H) 0.21 (s, $SiMe_3$), 4.56 (s, SiH), 7.49–7.92 (m, Ar); δ(F) −63.4; δ(Si) 0.04 (s, $SiMe_3$), −30.85 (t, SiH_2, ^1J(SiH) 199.0, ^3J(SiH) (to *o*-H) 5.1 Hz).
$TsiSiH_2F$	δ(H) 0.215 (d, $SiMe_3$, ^5J(HF) 0.5 Hz), 4.08 (d, SiH, ^2J(HF 52.0 Hz); δ(F) −178.49 (t, ^2J(HF) 51.9 Hz); δ(Si) −1.33 (d, $SiMe_3$, ^3J(SiF) 3.6 Hz), −5.02 (dd, SiH_2F, ^1J(SiF) 294.0; ^1J(SiH), 227.8 Hz).
TsiSiHBuF	δ(H) 0.215 (d, $SiMe_3$, ^5J(HF) 0.5Hz), 0.80–1.59 (m, Bu), 4.8 (d of multiplets; doublet ^2J(HF) 51.0 Hz), SiH); δ(F) −161.85 (d of multiplets, ^2J(HF) 51.0 Hz); δ(Si) −1.86 (d, $SiMe_3$, ^3J(SiF) 4.2 Hz), 16.21 (m, SiHFBu, ^1J(SiF) 293.9, ^1J(SiH) 215.1, ^2J(SiH) 4.7 Hz).

[a] Solutions in $CDCl_3$; ^{19}F shifts are relative to external $CFCl_3$, and ^{29}Si shifts relative to external $SiMe_4$.

in hexane (0.42 cm^3) and the mixture was stirred at room temperature for 2 h. Work-up as in (a) gave a white solid residue; this was shown by GLC/MS and ^1H NMR spectroscopy to be virtually exclusively $TsiSiH_2Bu$, which was isolated in ca. 60% yield by sublimation under vacuum.

2. *Reactions of $TsiSiH_2I$*

A procedure similar to that described under 1(a) for the reaction of $TsiSiH_3$ (for details see Table 1) gave the results indicated in Table 1.

In the case of *p*-$MeOC_6H_4Li$, the reaction was repeated with the organolithium reagent formed from the bromide and small pieces of freshly cut lithium in ether.

3. *Reactions of* $TsiSiH_2F$

Procedures similar to that described under 1(a), but starting from $TsiSiH_2F$ (see below) gave the results shown in Table 1. In a variation of the general procedure, after the reactions with p-$MeOC_6H_4Li$ and p-ClC_6H_4Li, the initial product mixture (after analysis) was recrystallized from EtOH before sublimation. (Found for $TsiSiH_2(C_6H_4Cl$-$p)$: C, 51.5; H, 9.1. $C_{16}H_{33}ClSi_4$ calc: C, 51.5; H, 8.9%.)

4. *Reactions of TsiSiHBuX (X = H or F)*

(a) A mixture of a solution of n-BuLi (1.05 mmol) in hexane (0.44 cm^3) with one of $TsiSiH_2Bu$ (0.75 mmol) in ether (5 cm^3) was refluxed for 1 h then stirred at room temperature for 2 h. The usual work-up left a liquid, which was judged from analysis by GLC/MS to contain $TsiSiHBu_2$ (60%), unchanged $TsiSiH_2Bu$ (20%), and small amounts of several other products (20% in total). Peaks in the ^1H NMR spectrum at δ 0.27 (s), 1.16 (m), and 3.88 (m) were assigned to $TsiSiHBu_2$.

(b) A mixture of PhLi (1.05 mmol) in 70/30 cyclohexane/Et_2O (0.6 cm^3) with a solution of $TsiSiH_2Bu$ (0.75 mmol) in ether (5 cm^3) was stirred at room temperature for 3 h. The usual work-up left a solid residue, which was judged from analysis by GLC/MS to consist of Ph_3SiBu (60%) and unchanged $TsiSiH_2Bu$ (40%). Recrystallization from EtOH gave Ph_3SiBu, m.p. 86 °C (lit., [7] m.p. 87.5–88.0 °C) (Found: C, 83.3; H, 8.0. $C_{22}H_{24}Si$ calc: C, 83.5; H, 7.6%); δ(H) 0.96 (t, 3H), 1.48 (m, 6H), and 7.45 (m, 15H).

(c) A mixture of BuLi (1.1 mmol) in hexane (0.46 cm^3) with a solution of TsiSiHBuF (1.0 mmol) in ether (7 cm^3) was stirred at room temperature for 4 h. The usual work-up gave a liquid residue, which was shown by GLC/MS analysis to contain $TsiSiHBu_2$ (90%) and unchanged TsiSiHBuF (10%). Peaks in the ^1H spectrum at δ 0.27(s), 1.12 (m), and 3.90 (m) were attributed to $TsiSiHBu_2$.

(d) A procedure similar to that in the preceding experiment, but starting with PhLi in 70/30 cyclohexane/Et_2O (0.65 cm^3) gave an oil, which was judged from GLC/MS analysis to consist of TsiSiHBuPh (90%) and unchanged TsiSiHBuF (10%); peaks at δ 0.32 (s), 1.12 (m), and 4.60 (m) p.p.m. were attributed to TsiSiHBuPh.

5. *Preparations of iodides, TsiSiHRI (R = Bu or* C_6H_4X*)*

(a) A mixture of $TsiSiH_2Bu$ (1.95 g, 6.12 mmol) in CCl_4 (20 cm^3) with a (1 *M*) solution of ICl (8 mmol) in CCl_4 (8 cm^3) was kept at room temperature for 5 h. The solvent was then evaporated off, and the residue washed several times with small amounts of CCl_4 to remove most of the remaining ICl, and the rest was volatalized off under vacuum. The residue was sublimed to give TsiSiHBuI (2.4 g, 88%), m.p. 50 °C; ^1H NMR data appear in Table 2).

(b) A (1 *M*) solution of ICl (2.0 mmol) in CCl_4 (2.0 cm^3) was added to a stirred solution of $TsiSiH_2(C_6H_4OMe$-$p)$ (2.0 mmol) in CCl_4 (12 cm^3) at room temperature. After 5 min the solvent was evaporated off under vacuum, the residue was washed several times with small amounts of CCl_4, and the residue sublimed under vacuum to give $TsiSiH(C_6H_4OMe$-$p)I$ (60%).

A similar procedure, but with reaction times of 10–20 min, gave the other $TsiSiH(C_6H_4Y)I$ compounds with Y = H (80%), p-Me (85%), or p-CF_3 (70%); NMR data are shown in Table 2.

6. Preparations of fluorides, TsiSiHRF (R = H or Bu)

(a) Silver tetrafluoroborate (5.5 mmol) was added to a solution of $TsiSiH_2I$ (5.0 mmol) in ether (20 cm^3) and the suspension was stirred at room temperature for 10 min. Filtration, followed by evaporation of the solvent, and sublimation (under vacuum) of the residue gave $TsiSiH_2F$ (80%). Details of its NMR spectra are given in Table 2.

(b) To a suspension of $AgBF_4$ (5.2 mmol in ether (20 cm^3) was added TsiSiH(Bu)I (5.0 mmol). The mixture was stirred for 1 h at room temperature then filtered, and the filtrate was evaporated. The residue was washed with a little MeOH then dried under vacuum and shown to be TsiSiH(Bu)F, m.p. ca. 40 °C. Details of its NMR spectra are given in Table 2.

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