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Dibisyl(fluorenylidene)stannene: evidence of its formation

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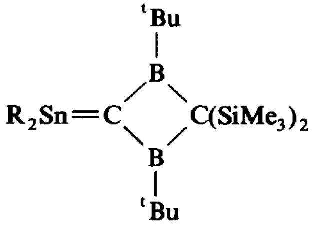
Abstract

The dibisyl(fluorenylidene)stannene **4**, obtained by dehydro-chlorination or -fluorination of the corresponding chloro- or fluoro-stannanes **5** and **6** by tert-butyllithium is an extremely air-sensitive compound. It has not been isolated, but identified by trapping reactions by lithio compound and water.

Introduction

Low-coordinated species of group 14 elements have been intensively studied for a few years. Many doubly-bonded compounds of silicon $>$Si=X (X = C, Si, N, P, S, Se) have now been isolated [1]. Derivatives with low-coordinated germanium are much less numerous [2], but some of them have been prepared and isolated as monomers, particularly some germenes $>$Ge=C$<$ [3,4], digermenes $>$Ge=Ge$<$ [5], germa-imines $>$Ge=N– [6] and germaphosphenes $>$Ge=P– [7] and their reactions are well known. In contrast doubly-bonded tin derivatives are rare. The stabilization of monomeric species is, of course, more difficult given the heavier elements in a group, because, among other reasons, of poorer orbital overlap. The same effect is observed in other groups, for example group 15; many doubly-bonded nitrogen and phosphorus compounds have been synthesized, but it is much more difficult to isolate low coordinated arsenic and, particularly, antimony compounds.

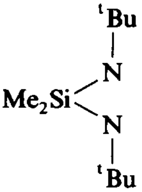
Thus up to now only three doubly-bonded tin derivatives at room temperature in solution have been observed, namely stannenes **1a** and **1b** isolated by Berndt [3b,8] and the stannaphosphene **2** [9] prepared by us:



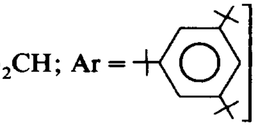
(1a, 1b)

$Bis_2Sn{=}PAr$

(2)

$R_2 = Bis_2$, **1a**;  **1b**

$\mathrm{Me_2Si(N{}^tBu)_2}$

Bis = $(Me_3Si)_2CH$; Ar = 

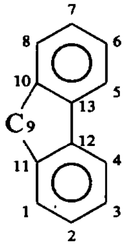
A distannene $Ar'_2Sn{=}SnAr'_2$ (Ar' = 2,4,6-tri-isopropylphenyl) with a Sn=Sn structure has been observed in solution by Masamune [10], but above 0 °C it gives the cyclotristannane $(Ar'_2Sn)_3$ via stannylene addition to the tin–tin double bond. The dibisyldistannene $Bis_2Sn{=}SnBis_2$ made by Lappert reacts in solution as two stannylenes Bis_2Sn and has a distannene structure only in the solid state [11]. This behaviour is typical for a heavy element such as tin, which has a great tendency to give stannylenes.

Analogues of alkenes involving heavier elements of group 14 should be of much interest in organic and in organometallic synthesis (preparation of heterocyclic and functionalized derivatives, etc) or as potential precursors of new types of polymers. But though silenes and germenes have been extensively studied, very little is known about stannenes except for the addition of HCl to the tin–carbon double bond of **1** [8], and the reaction of a transient stannene with phenyllithium, dimethylbutadiene, and a silyl azide [12].

Following our studies of germenes **3** [4], we decided to try to prepare the stannene **4**, bearing two bis(trimethylsilyl)methyl (bisyl) groups on tin and with the relevant carbon included in the fluorenylidene moiety.

$Bis_2Sn{=}CR_2$

(**4**)

Bis = $(Me_3Si)_2CH$; CR_2 = 

$R'_2Ge{=}CR_2$

(**3**)

$R'_2 = Mes_2$; $(R_2CH)_2$; $((Me_3Si)_2CH)_2$

R_2CH, ^tBu; $(Me_3Si)_2CH$, Mes

Such a compound should be stabilized, like the germenes **3**, by bulky groups on tin and conjugative interaction between the tin–carbon double bond and the fluorenylidene group.

Results and discussion

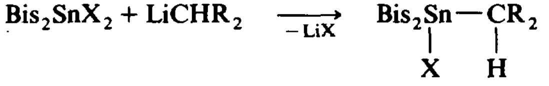
The best route to unsaturated main group species is usually dehydrochlorination (or dehydrofluorination) by organolithium compounds of chloro (or fluoro) precursors: for example, germenes $>Ge{=}C<$ [4], germaphosphenes $>Ge{=}P-$ [7], and a stannaphosphene $>Sn{=}P-$ [9] have been prepared in this way. Thus the first step in the synthesis of **4** involved the preparation of chloro(fluorenyl)stannane (**5**) and of fluoro(fluorenyl)stannane (**6**), by reaction between the fluorenyllithium and the

Table 1

NMR data (solvent $CDCl_3$) for stannanes

		5	6	9	11	14	15	20
^1H	CH	–	–	−0.46 (s)	0.23 (s)	−0.32 (d) ^3J(HH) = 1.5	–	−0.22 (s)
	Me_3Si	−0.01 (s) 0.10 (s)	0.44 (s) 0.62 (s)	−0.05 (s) 0.09 (s)	0.02 (s) 0.13 (s)	0.04 (s) 0.22 (s)	−0.02 (s) 0.14 (s)	0.05 (s) 0.22 (s)
	H(Me)	–	–	Me: 0.42 (s)	–	6.04 (t) ^3J(HH) = 1.5	2.03 (s)	–
	CH	4.83 (s)	5.11 (s)	4.65 (s)	4.88 (s)	4.59 (s)	–	4.67 (s)
	CR_2	7.14–8.09 (m)	7.47–8.29 (m)	7.17–8.06 (m)	7.17–8.06 (m)	6.94–7.97	7.21–8.13 (m)	6.95–7.92 (m)
^{13}C	CH	11.74	–	2.28	9.92	2.60	–	9.78 (^1J(SnC) = 184.8)
	Me_3Si	3.46 4.25	–	3.51 4.32	3.55 4.35	3.17 3.56	–	3.44 (^3J(SnC) = 18.1) 4.03 (^3J(SnC) = 18.3)
	CH(Me)	48.55	–	42.90 Me: −0.45	46.49	41.5	–	47.70 (^3J(SnC) = 240.3)
	C_4C_5	120.30	–	120.16	120.23	120.07	–	120.48
	C_1C_8	124.80	–	124.00	124.74	124.30	–	124.08 (^3J(SnC) = 18.1)
	$C_2C_3C_6C_7$	126.09 126.69	–	124.78 126.06	126.09 126.55	125.02 126.28	–	125.61, 126.42 (^4J, ^5J(SnC) = 8)
	$C_{12}C_{13}$	140.42	–	139.83	140.25	139.80	–	140.10 (^3J(SnC) = 5)
	$C_{10}C_{11}$	144.09	–	146.80	144.64	146.86	–	144.63 (^2J(SnC) = 15.0)
^{119}Sn (^2J(SnH))		99.5 (q) (96)	89.7 (dq) (100) ^1J(SnF) = 2563	25.9 (dtq) (81.8, 51.3, 44.9)	−0.4 (q) (90)	−29.4 (dq) (86) ^1J(SnH) = 1797	−13.6 (dq) (< 10 Hz) ^3J(SnH) = 77	48.3 (q) (82)
^{19}F	–	–	−110.0	–	–	–	–	–

corresponding dihalogenostannanes **7**, **8**:

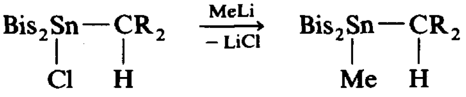


$$\mathrm{Bis_2SnX_2 + LiCHR_2} \xrightarrow[-\mathrm{LiX}]{} \underset{\mathrm{X}}{\mathrm{Bis_2Sn}}-\underset{\mathrm{H}}{\mathrm{CR_2}}$$

(X = Cl, **7**; F, **8**) (X = Cl, **5**; F, **6**)

Compounds **5** and **6** were characterized by NMR (see Table 1) and mass spectroscopy. As expected in the ^1H and ^{13}C NMR spectra two signals are observed for the trimethylsilyl groups: because of the prochirality of tin, the two Me_3Si within each bisyl group are inequivalent; such an effect was noted previously by Lappert for the related compounds $Bis_2Sn(X)Y$ [13].

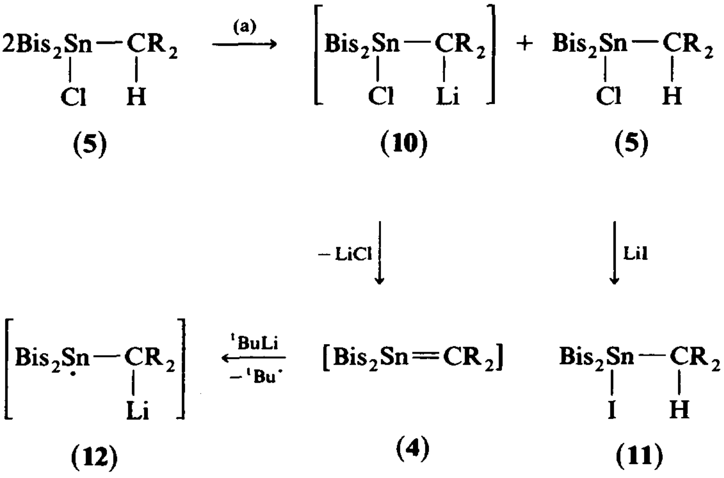
The reaction of **5** with methyllithium did not afford the expected stannene **4**, but solely compound **9** (see Table 1) owing to direct alkylation of tin by the lithio-compound:

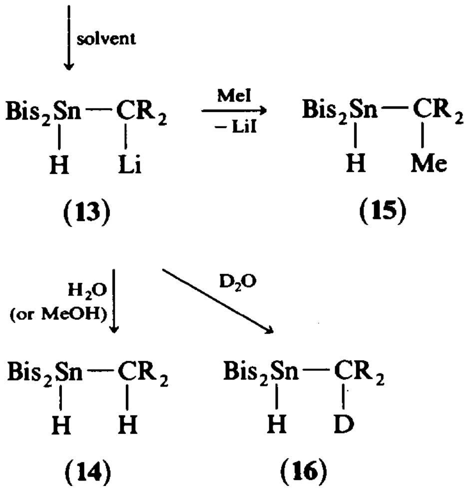


$$\underset{\mathrm{Cl}}{\mathrm{Bis_2Sn}}-\underset{\mathrm{H}}{\mathrm{CR_2}} \xrightarrow[-\mathrm{LiCl}]{\mathrm{MeLi}} \underset{\mathrm{Me}}{\mathrm{Bis_2Sn}}-\underset{\mathrm{H}}{\mathrm{CR_2}}$$

(**5**) (**9**)

It thus seemed that a bulkier organolithium compound, such as tert-butyllithium would have to be used in order to avoid such alkylation of tin; the addition of this lithio compound was performed at low temperature, then the mixture was quenched at 20 °C with methanol, D_2O, or methyl iodide. After methanolysis the NMR spectroscopic analysis showed the formation of the stannane **14** along with the starting chlorostannane **5** in a 50/50 ratio. Quenching with D_2O afforded the deuterofluorenylstannane **16**, and again some of the starting chlorostannane **5**. With methyl iodide, the methylfluorenylstannane **15** was obtained along with iodostannane **11**: the surprising formation of **11** probably arises from the corresponding chloro species by exchange with lithium iodide formed in the reaction mixture. The formation of all the products, **11**, **14**, **15** and **16** (see NMR data in Table 1), can be accounted for in terms of the following reaction scheme:





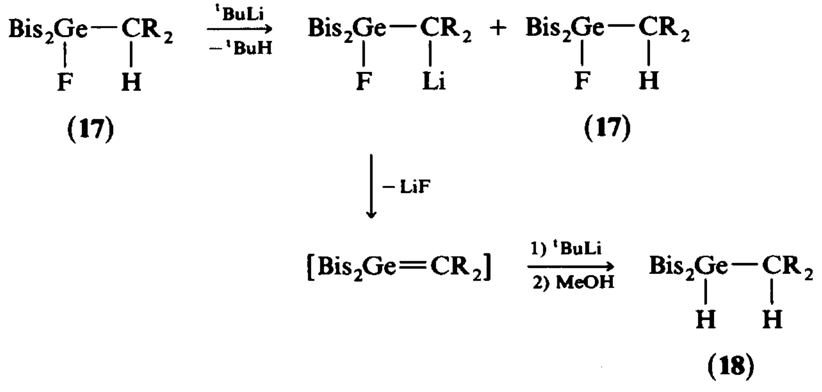
$$\Big\downarrow{\scriptstyle\text{solvent}}$$

$$\underset{(13)}{\mathrm{Bis_2}\underset{\mathrm{H}}{\mathrm{Sn}}-\underset{\mathrm{Li}}{\mathrm{CR_2}}} \xrightarrow[-\mathrm{LiI}]{\mathrm{MeI}} \underset{(15)}{\mathrm{Bis_2}\underset{\mathrm{H}}{\mathrm{Sn}}-\underset{\mathrm{Me}}{\mathrm{CR_2}}}$$

$$\mathrm{(13)}\ \xrightarrow[\text{(or MeOH)}]{\mathrm{H_2O}}\ \underset{(14)}{\mathrm{Bis_2}\underset{\mathrm{H}}{\mathrm{Sn}}-\underset{\mathrm{H}}{\mathrm{CR_2}}} \qquad \mathrm{(13)}\ \xrightarrow{\mathrm{D_2O}}\ \underset{(16)}{\mathrm{Bis_2}\underset{\mathrm{H}}{\mathrm{Sn}}-\underset{\mathrm{D}}{\mathrm{CR_2}}}$$

It seems that the lithiation of **5** to form **10** is slow, whereas the elimination of lithium chloride from **10** to afford stannene (**4**) occurs very rapidly: so that the stannene is formed before complete consumption of tert-butyllithium, and reacts with this lithio-compound, by a single electron transfer, to give the transient **12** species, and then **13**; quenching of **13** with MeOH, MeI or D_2O, respectively, leads to **14**, **15** and **16**.

A similar reaction was observed in the synthesis of dibisylfluorenylidenegermene [4c]: reaction of tert-butyllithium with dibisylfluorofluorenylgermane (**17**) in pentane or diethyl ether followed by quenching with methanol gave unchanged **17** and the hydrogermane **18**, via the lithio-species $Bis_2Ge(H)C(Li)R_2$:



$$\underset{(17)}{\mathrm{Bis_2}\underset{\mathrm{F}}{\mathrm{Ge}}-\underset{\mathrm{H}}{\mathrm{CR_2}}} \xrightarrow[-{}^t\mathrm{BuH}]{{}^t\mathrm{BuLi}} \mathrm{Bis_2}\underset{\mathrm{F}}{\mathrm{Ge}}-\underset{\mathrm{Li}}{\mathrm{CR_2}} + \underset{(17)}{\mathrm{Bis_2}\underset{\mathrm{F}}{\mathrm{Ge}}-\underset{\mathrm{H}}{\mathrm{CR_2}}}$$

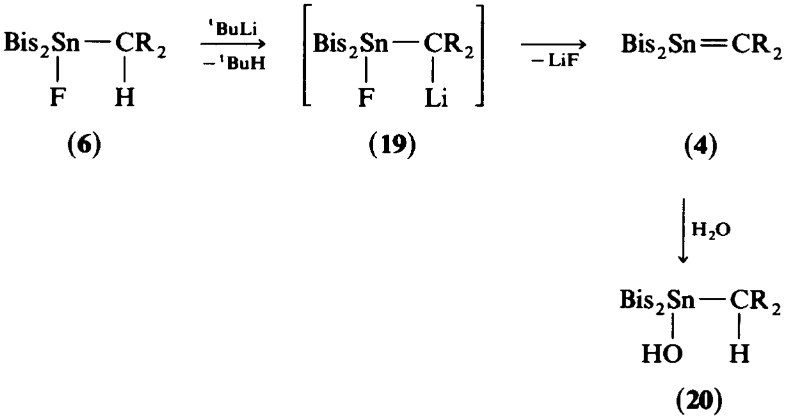
$$\Big\downarrow{\scriptstyle -\mathrm{LiF}}$$

$$[\mathrm{Bis_2Ge{=}CR_2}] \xrightarrow[\text{2) MeOH}]{\text{1) }{}^t\mathrm{BuLi}} \underset{(18)}{\mathrm{Bis_2}\underset{\mathrm{H}}{\mathrm{Ge}}-\underset{\mathrm{H}}{\mathrm{CR_2}}}$$

The germene was obtained in a pure form only by using a 50/50 Et_2O/THF mixture, which favours the formation of the lithiofluorogermane precursor.

In the case of **5** when the dehydrochlorination reaction was performed in Et_2O or in a Et_2O/THF mixture or even in pure THF, we always obtained the same result and it seems that the reaction leading to lithio compound **10** is still too slow compared to the elimination of lithium chloride.

Use of the fluorostannane **6** as the starting point provides a better solution to the synthesis of stannene: because of the strength of the Sn–F bond, elimination of lithium fluoride from the lithio-compound **19** occurs only at room temperature, after all the tert-butyllithium has reacted so that after hydrolysis we have never observed the formation of hydrostannane **14**. Despite use of such conditions, it has not been possible to isolate the expected stannene **4**: fractional crystallization gave only the hydroxystannane **20** (see Table 1), along with some unidentified products. It thus seems that the stannene **4** is extremely air- and moisture-sensitive, more so than the corresponding germene species [4], and thus only its hydrolysis product could be obtained:



$$\underset{\textbf{(6)}}{\underset{\mathrm{F}\qquad\mathrm{H}}{\mathrm{Bis_2Sn{-}CR_2}}} \xrightarrow[-{}^t\mathrm{BuH}]{{}^t\mathrm{BuLi}} \underset{\textbf{(19)}}{\left[\underset{\mathrm{F}\qquad\mathrm{Li}}{\mathrm{Bis_2Sn{-}CR_2}}\right]} \xrightarrow[-\mathrm{LiF}]{} \underset{\textbf{(4)}}{\mathrm{Bis_2Sn{=}CR_2}} \xrightarrow{\mathrm{H_2O}} \underset{\textbf{(20)}}{\underset{\mathrm{HO}\qquad\mathrm{H}}{\mathrm{Bis_2Sn{-}CR_2}}}$$

We are now using bulkier substituents on tin in an attempt to enhance the stability of compounds with a tin–carbon double bond.

Experimental

General data

All the syntheses were performed by high-vacuum line techniques and with carefully deoxygenated solvents which were freshly distilled over sodium-benzophenone. ^{1}H NMR spectra were recorded on Bruker AC 80 and AC 200 spectrometers at 80.1 and 200.1 MHz, respectively. ^{13}C NMR spectra were recorded on Bruker AC 200 and AC 250 instruments at 50.3 and 62.9 MHz (TMS internal standard), and ^{119}Sn NMR spectra on Bruker AC 200 instrument at 74.6 MHz (Me_4Sn internal standard). IR spectra were recorded on a Perkin Elmer 1600 (FT) spectrometer. Mass spectra were obtained with a Nermag R10 010 spectrometer by EI at 70 eV or by DCI with CH_4. Melting points were determined with a Reichert apparatus. Elemental analyses were performed by the "Service de micro analyse de l'Ecole de Chimie de Toulouse" (France). The carbon atoms of the fluorenyl group are numbered C_1 to C_{13} according to the diagram in the Introduction.

Synthesis of dibisyl(fluorenyl)chlorostannane (5)

A solution of fluorenyllithium (prepared from fluorene (1.92 g, 11.57 mmol), 7.25 ml of butyllithium 1.6 *M* in hexane and 25 ml of Et_2O) was slowly added to a solution of bis[bis(trimethylsilyl)methyl]dichlorostannane (5.86 g, 11.54 mmol) in Et_2O (30 ml) at −10 °C; the red colour of the fluorenyllithium immediately disappeared. The mixture was allowed to warm to room temperature and was filtered to remove LiCl; the solvents were removed *in vacuo* and the crude **5** was recrystallized from pentane to give 6.1 g (83%) of white crystals of **5**, m.p. = 137–138 °C. MS (DCI/CH_4, ^{120}Sn): *m/e* 623 ($M^+ + 1 - CH_4$, 87), 603 ($M^+ + 1 - HCl$, 39), 473 ($Bis_2SnCl + 1$, 100). Analysis: Found: C, 50.20; H, 7.34. $C_{27}H_{47}ClSi_4Sn$ calcd.: C, 50.85; H, 7.37%.

Synthesis of dibisyl(fluorenyl)fluorostannane (6)

The fluoroderivative **6** was made by the same procedure but from 4.12 g of dibisyldifluorostannane **8** [9] (8.68 mmol) and one equivalent of fluorenyllithium (prepared from fluorene (1.44 g, 8.68 mmol), 5.42 ml of butyllithium 1.6 *M* in hexane and 30 ml Et_2O). Recrystallization in pentane gave 4.04 g of white crystals of **6** (75%), m.p.: 168–169 °C.

Analysis: Found: C, 52.35; H, 7.64. $C_{27}H_{47}FSi_4Sn$ calcd.: C, 52.20; H, 7.57%. MS (EI, 70 eV, ^{120}Sn): 622 (*M*, 3), 607 (*M* – 15, 10), 457 (Bis_2SnF, 80), 165 (CHR_2, 100).

Reaction of 5 with tert-butyllithium

To a solution of **5** (1.05 g, 1.65 mmol) in Et_2O (10 ml) cooled at -78 °C was added 0.97 ml (1.65 mmol) of tert-butyllithium 1.7 *M* in pentane. The mixture , which turned orange, was warmed to room temperature, stirred for 1 h, and quenched with methanol. After filtration to remove LiCl and MeOLi, the ^1H and ^{119}Sn NMR spectra of the solution showed the presence of **14** and of the starting material **5** in a 50/50 ratio. Recrystallization in Et_2O afforded **5** then **14**; **14**: 0.48 g (48%) white crystals, m.p.: 145 °C. IR: $\nu(SnH)$: 1845 cm^{-1}.

Quenching of 13 by MeI and D_2O

(a) MeI: A solution of tert-butyllithium in pentane (0.94 ml, 1.60 mmol) was added from a syringe to a solution of **5** (1.02 g, 1.60 mmol) in Et_2O (10 ml) at -78 °C. The mixture was allowed to warm to room temperature then an excess of methyl iodide was added to the orange solution (of **13**). A slow decoloration was observed. The solid material was removed by filtration. The ^1H and ^{119}Sn NMR spectra of the solution showed the presence of iodostannane **11** and the expected methylfluorenylstannane **15**.

15: NMR ($CDCl_3$). ^1H: -0.02 (s, 18H, Me_3Si); 0.14 (s, 18H, Me_3Si); 2.03 (s, 3H, CMe); 7.21–8.13 (m, 8H, CR_2). ^{119}Sn: -13.6 ($^1J(SnH) = 1692$, $^2J(SnH) < 10$, $^3J(SnH) = 77$ Hz). Compound **15** showed poor stability and decomposed within a few hours in solution. Successive recrystallizations afforded fluorenylmethane $R_2C(H)Me$ [0.14 g, 49%, NMR ($CDCl_3$. ^1H: 1.66 (d, $^3J(HH) = 0.5$ Hz, 3H, Me); 2.06 (q, $^3J(HH) = 0.5$ Hz, 1H, CH); 7.18–7.64 (m, 8H, CR_2)] and dibisyl(fluorenyl)iodostannane **11** (0.59 g, 51%, m.p.: 169–170 °C). MS (DCI/CH_4, ^{120}Sn): *m/e* 715 ($M^+ + 1 - CH_4$, 76), 603 ($M^+ + 1 - HI$, 42), 565 (Bis_2SnI, 100), 438 ($Bis_2Sn + 1$, 11)].

(b) D_2O: Quenching of orange solution of **13** prepared as described above with D_2O afforded a colorless mixture. NMR analysis showed the presence of the deuterocompound **16**: $\delta(^{119}Sn)$: -27.5 (dt, $^1J(SnH) = 1790$, $^2J(SnH) = 87$ Hz).

Reaction of methyllithium with 5

To a solution of **5** (1.04 g, 1.63 mmol) in Et_2O (10 ml) at -78 °C was added one equivalent of 1.6 *M* methyllithium in Et_2O (1.02 ml). The mixture remained colorless. LiCl was removed by filtration and the Et_2O was evaporated of *in vacuo*. Recrystallization of crude **9** from pentane afforded 0.92 g (92%) of white crystals;

m.p.: 125–126 °C. MS (DCI/CH_4, ^{120}Sn): *m/e* 603 ($M^+ + 1 - CH_4$, 57), 453 ($Bis_2SnMe + 1$, 100). Analysis: Found: C, 54.47; H, 8.22. $C_{28}H_{50}Si_4Sn$ calcd.: C, 54.48; H, 8.11%.

*Reaction of fluorostannane **6** with tert-butyllithium*

To a solution of fluorostannane **6** (1.06 g, 1.71 mmol) in Et_2O (10 ml) at −78 °C was added one equivalent of 1.7 *M* tert-butyllithium in pentane. The mixture immediately turned orange. After 2 h stirring at room temperature methanol was added and the solution filtered. The ^{119}Sn NMR of spectrum the solution showed the presence of starting **6** and of three unidentified products giving signals δ $^{119}Sn = 48.3$, 33.4 and −9.8. The hydroxystannane **20** δ: 48.25) was isolated by recrystallization from pentane; (0.20 g (19%) of white crystals). IR: ν(OH) 3620 cm^{-1}. MS (EI, 70 eV, ^{120}Sn): 455 (Bis_2SnOH, 20), 440 ($Bis_2SnOH - Me$, 15), 165 (CHR_2, 100) Analysis: Found: C, 52.11; H, 7.89. $C_{27}H_{48}OSi_4Sn$ calcd.: C, 52.33; H, 7.81%.

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