

# Chemistry of the stable diarylstannylene ( $R_F$ )<sub>2</sub>Sn ( $R_F \equiv$ nonafluoromesityl): oxidative addition reactions<sup>1</sup>

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## Abstract

The preparation of various bis(nonafluoromesityl)tin(IV) derivatives starting from ( $R_F$ )<sub>2</sub>Sn (**1**) is described ( $R_F \equiv$  2,4,6-tris(trifluoromethyl)phenyl, "nonafluoromesityl"). The fluorination of **1** with XeF<sub>2</sub> affords the corresponding difluoride ( $R_F$ )<sub>2</sub>SnF<sub>2</sub> (**2**). The dichloro species ( $R_F$ )<sub>2</sub>SnCl<sub>2</sub> (**4**) has been prepared via a new synthetic route by the treatment of **1** with SnCl<sub>4</sub>. Compound **4** serves as a useful precursor for the synthesis of dibenzoates of the type ( $R_F$ )<sub>2</sub>Sn(O<sub>2</sub>CC<sub>6</sub>H<sub>4</sub>R-*o*)<sub>2</sub> (**5**, R ≡ H; **6**, R ≡ F). Insertion of **1** into the sulphur–sulphur or selenium–selenium bond of aromatic dichalcogenides provides the tin(IV) bis(chalcogenolates) ( $R_F$ )<sub>2</sub>Sn(EC<sub>6</sub>H<sub>4</sub>R<sub>3</sub>-2,4,6)<sub>2</sub> (**7**, E ≡ S, R ≡ Me; **8**, E ≡ Se, R ≡ CF<sub>3</sub>). All new compounds have been fully characterized by spectroscopic methods and elemental analyses.

**Keywords:** Diarylstannylene; Nonafluoromesityl; Oxidative addition reactions

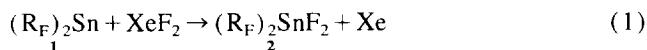
## 1. Introduction

During the past 10 years, the 2,4,6-tris(trifluoromethyl)phenyl ("nonafluoromesityl", abbreviated as  $R_F$ ) substituent has been demonstrated to be a highly versatile building block in main group chemistry [1–4]. Due to its ideal combination of sterically and electronically stabilizing effects, this substituent has been successfully employed in the stabilization of low coordination numbers around various main group elements, such as phosphorus [5], tin [6], lead [7], indium [8] and thallium [9]. Of special interest are the readily accessible and unusually stable nonafluoromesityl derivatives of divalent tin and lead [3,6,7]. ( $R_F$ )<sub>2</sub>Pb was the first diarylplumbylene ever made. However, its derivative chemistry has thus far been limited to very few reactions involving the replacement of the organic ligands [3,7]. Much more interesting and diverse is the chemistry of the stable diarylstannylene derivative ( $R_F$ )<sub>2</sub>Sn (**1**). Here we report several new reactions of **1** leading to bis(nonafluoromesityl)tin(IV) species containing additional halide, carboxylate or chalcogenolate substituents.

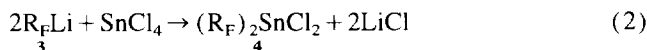
## 2. Results and discussion

In contrast with its lead(II) homologue ( $R_F$ )<sub>2</sub>Pb, the stable tin(II) derivative bis[2,4,6-tris(trifluoromethyl)phenyl]tin

( $R_F$ )<sub>2</sub>Sn (**1**) readily undergoes oxidation reactions to afford the corresponding tin(IV) species [3]. For example, treatment of **1** with AsF<sub>5</sub> yields ( $R_F$ )<sub>2</sub>SnF<sub>2</sub> (**2**) [10], while the dichloride ( $R_F$ )<sub>2</sub>SnCl<sub>2</sub> (**4**) can be prepared by chlorinating **1** with elemental chlorine. In addition to these previously described halide addition reactions, we found that the fluorination of **1** using XeF<sub>2</sub> in acetonitrile solution provides excellent access to bis(nonafluoromesityl)tin(IV) difluoride **2**:



A more reliable synthetic route to ( $R_F$ )<sub>2</sub>SnCl<sub>2</sub> involves the reaction of tin(IV) chloride with two equivalents of  $R_F\text{Li}$  (**3**) in diethyl ether and affords the product in 68% yield:

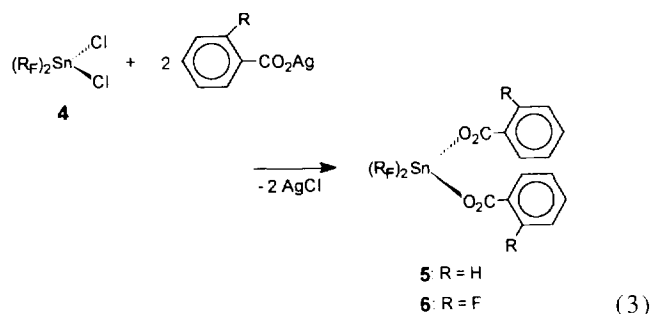


The dichloride **4** has been characterized by spectroscopic methods and elemental analysis. It serves as a useful starting material for the preparation of hitherto unknown carboxylate derivatives containing the ( $R_F$ )<sub>2</sub>Sn moiety. Both chlorine atoms are easily replaced by benzoate anions when **4** is reacted with two equivalents of silver benzoates according to reaction (3). For the success of these preparations, it is important to use carefully dried acetonitrile as solvent. No reaction is observed in hydrocarbon solvents, whereas tetrahydrofuran (THF) is polymerized under the reaction conditions. Similar reactions using alkali metal carboxylates as reagents did not

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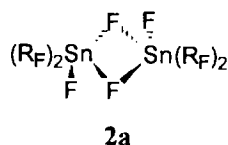
<sup>1</sup> Dedicated to Professor Erwin Weiß on the occasion of his 70th birthday.

afford crystalline reaction products. The two tin(IV) benzoates **5** and **6** have been obtained in moderate yields as white, microcrystalline solids.

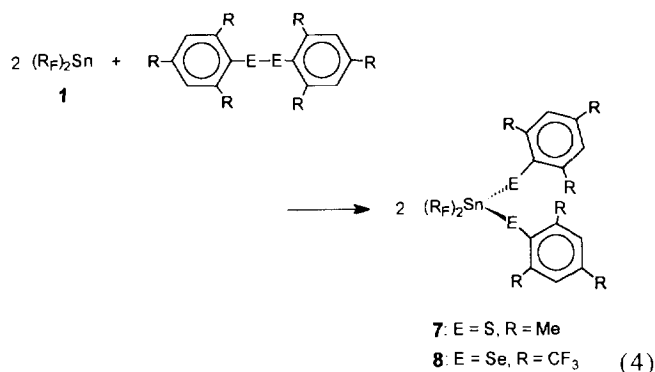


During the course of this work, a careful analysis of the IR spectra of **2**, **4**, **5** and **6**, as selected members of the  $R_F$  series, was carried out in order to confirm the proposed compositions. Bands in the ranges of  $1626\text{--}1617\text{ cm}^{-1}$  and  $695\text{--}691\text{ cm}^{-1}$  are typical for the valence and deformation vibrations of the  $\text{CF}_3$ - and carboxy-substituted phenyl rings, respectively. In the case of **6**, the shoulder at  $1626\text{ cm}^{-1}$  can be attributed to the nonafluoromesityl group and the stronger band at  $1617\text{ cm}^{-1}$  originates from the benzoate groups. In the ranges  $1302\text{--}1265$ ,  $1199\text{--}1131$  and  $687\text{--}668\text{ cm}^{-1}$ , the respective valence and deformation vibrations of the  $\text{CF}_3$  groups can be detected. In the spectrum of **6**, the band at  $1235\text{ cm}^{-1}$  is attributed to the C–F vibration of the *o*-fluorobenzoate anion. The  $\nu_{\text{as}}(\text{COO}^-)$  bands are observed at  $1603\text{ cm}^{-1}$  (**5**) and  $1599\text{ cm}^{-1}$  (**6**) respectively, whereas the  $\nu_{\text{s}}(\text{COO}^-)$  band is found at  $1420\text{ cm}^{-1}$ . The latter assignment was confirmed by the Raman spectrum of **6**.

In the IR spectrum of **2**, the absorption at  $489\text{ cm}^{-1}$  is attributed to the Sn–F vibration. For  $\text{Cl}_2\text{SnF}_2$ , the  $\nu_{\text{s}}(\text{SnF}_2)$  band is observed at a wavenumber  $64\text{ cm}^{-1}$  lower than  $\nu_{\text{as}}(\text{SnF}_2)$ , and for  $\text{SnF}_2(\text{SO}_3\text{F})_2$  the difference is  $79\text{ cm}^{-1}$  [11,12]. Neither the IR or Raman spectrum of **2** exhibits a comparable vibration for  $\nu_{\text{s}}(\text{SnF}_2)$ . Instead, a band at  $321\text{ cm}^{-1}$  is observed, which is indicative of an Sn–F–Sn bridging unit. In the spectra of methyltin(IV) fluorides, such fluorine bridges give rise to very broad strong bands in the range  $335\text{--}425\text{ cm}^{-1}$  [12]. On the basis of these IR spectroscopic results, a dimeric structure (**2a**) is postulated for the bis(nonafluoromesityl)tin(IV) difluoride **2**.



The stannylene **1** was also found to insert readily into the sulphur–sulphur or selenium–selenium bond of aromatic dichalcogenides to afford the corresponding tin(IV) dichalcogenolates (Eq. (4))



This reaction has been carried out with dimesityldisulphide and bis[2,4,6-tris(trifluoromethyl)]diselenide. Both products **7** and **8** have been isolated in the form of yellow crystals and characterized by spectroscopic methods.

It has been shown in this study that tin(IV) derivatives containing nonafluoromesityl substituents are easily accessible via two synthetic routes. The first involves oxidative addition reactions of  $(R_F)_2\text{Sn}$  (**1**), while the second uses  $(R_F)_2\text{SnCl}_2$  (**4**) as a precursor for metathetical reactions. Both methods complement each other and, by using either one of these routes, a large number of related bis(nonafluoromesityl)tin(IV) compounds should be available, e.g. for testing potential antitumour activities.

### 3. Experimental details

All reactions involving **1** or **4** were carried out under dry, purified nitrogen. Solvents were dried with Na/benzophenone and freshly distilled under  $\text{N}_2$  prior to use. IR spectra: Bio-Rad FTS 7 and Nujol/KBr. Nuclear magnetic resonance (NMR) spectra: Bruker WP 80 SY and Bruker 250 M. Elemental analyses: analytical laboratories of the Department of Inorganic Chemistry at the University of Göttingen and the Department of Chemistry at Magdeburg. The starting materials nonafluoromesitylene [5],  $R_F\text{Li}$  [1],  $(R_F)_2\text{Sn}$  (**1**) [6],  $\text{XeF}_2$  [13], dimesityldisulphide [14] and bis(nonafluoromesityl)diselenide [15] were prepared according to literature procedures. The silver benzoates  $\text{AgO}_2\text{CC}_6\text{H}_4\text{R-}o$  ( $R \equiv \text{H, F}$ ) [16] were prepared following a procedure described for anhydrous silver triflate [17]. These preparations are briefly described here.

#### 3.1. Silver benzoate ( $\text{AgO}_2\text{CC}_6\text{H}_5$ )

A suspension of 2.94 g (12.7 mmol) silver(I) oxide and 3.00 g (24.5 mmol) benzoic acid in pyridine (80 ml) was stirred for 24 h at room temperature. Unreacted  $\text{Ag}_2\text{O}$  was filtered off, the filtrate was evaporated to dryness with the exclusion of light and the remaining white solid was thoroughly (2 days) dried in vacuo to afford 5.79 g (24.5 mmol, 100%) of silver benzoate.  $\text{C}_7\text{H}_5\text{AgO}_2$  (229.0): calculated:

C, 36.6%; H, 2.2%; Ag, 47.1%; found: C, 36.5%; H, 1.9%; Ag, 47.1%.

### 3.2. Silver *o*-fluorobenzoate ( $\text{AgO}_2\text{CC}_6\text{H}_4\text{F-}o$ )

A sample of 3.72 g (12.5 mmol, 98%) of moderately light-sensitive silver *o*-fluorobenzoate was obtained in an analogous manner from 1.53 g (6.6 mmol)  $\text{Ag}_2\text{O}$  and 1.78 g (12.7 mmol) *o*-fluorobenzoic acid.  $\text{C}_7\text{H}_4\text{AgFO}_2$  (247.0): calculated: C, 34.0%; H, 1.6%; Ag, 43.7%; found: C, 34.5%; H, 1.9%; Ag, 45.0%.

### 3.3. Bis[2,4,6-tris(trifluoromethyl)phenyl]tin(IV) difluoride (2)

$\text{XeF}_2$  (0.35 g, 2.1 mmol) was added at  $-20^\circ\text{C}$  to a rapidly stirred solution of **1** (1.36 g, 2.0 mmol) in 30 ml of dry acetonitrile. After gas evolution had ceased, the reaction mixture was allowed to warm to room temperature and evaporated to dryness. The solid residue was recrystallized from boiling heptane (60 ml) to afford 1.27 g (88%) of **2** as colourless crystals. NMR spectroscopic data were in good agreement with those reported in the literature [10]. IR ( $\text{cm}^{-1}$ ): 3092 w, 3040 w, 3005 w, 1626 m, 1618 m, 1598 w, 1583 w, 1542 w, 1498 w, 1466 w, 1459 w, 1420 w, 1388 w, 1300 vs, 1265 s, 1199 vs, 1157 vs sh, 1157 vs, 1122 s sh, 1088 m, 915 m, 857 m, 840 w, 825 w, 755 w, 745 w, 695 m, 687 m, 670 m, 559 w, 489 m, 434 w, 406 w, 367 w, 321 w, 252 w, 224 m, 210 m.

### 3.4. Bis[2,4,6-tris(trifluoromethyl)phenyl]tin(IV) dichloride (4)

$\text{SnCl}_4$  (6.51 g, 25.0 mmol) was added dropwise at  $0^\circ\text{C}$  to 100 ml of diethyl ether. A suspension of the insoluble etherate was formed in an exothermic reaction. On slow addition of a freshly prepared solution of  $\text{R}_\text{F}\text{Li}$  (50.0 mmol) in 200 ml diethyl ether, the reaction mixture turned dark brown. The solution was heated under reflux for 48 h. Evaporation to dryness left a dark residue, which was redissolved in 200 ml of toluene and filtered through a thin layer of Celite filter-aid. The filtrate was concentrated to a total volume of 50 ml and diluted with 100 ml of hexane. Cooling to  $-20^\circ\text{C}$  for 48 h afforded pale yellow crystals of the product, which were isolated by filtration and dried under vacuum. Yield: 12.73 g (16.9 mmol, 68%), m.p.  $128^\circ\text{C}$ .

IR ( $\text{cm}^{-1}$ ): 3092 m (C–H arom.), 2280 w, 2229 w, 1843 m, 1626 s, 1580 m, 1343 s ( $\text{CF}_3$ ), 1302 s, 1281 s, 1269 s sh, 1203 s br, 1154 s br, 1089 s sh, 974 m br, 930 m, 915 s, 855 s, 840 s ( $\text{R}_\text{F}$  out-of-plane), 737 s, 722 s, 691 s, 685 s, 676 m, 559 m, 514 m, 433 s, 402 s, 376 vs (Cl–Sn–Cl asym.), 364 s. Electron impact mass spectrometry (EI-MS)  $m/z$  (%): 733 ( $\text{M}^+ - \text{F}$ , 8), 717 ( $\text{M}^+ - \text{Cl}$ , 18), 471 ( $\text{M}^+ - \text{R}_\text{F}$ , 100), 281 ( $\text{R}_\text{F}^+$ , 12), 262 ( $\text{R}_\text{F}^+ - \text{F}$ , 46), 243 ( $\text{R}_\text{F}^+ - 2\text{F}$ , 60), 153 ( $\text{SnCl}^+$ , 18), 69 ( $\text{CF}_3^+$ , 13).  $^1\text{H NMR}$  ( $\text{C}_6\text{D}_6$ , 250 MHz)  $\delta$ : 7.88 (s, 4H,  $\text{C}_6\text{H}_2$ ) ppm.  $^{19}\text{F NMR}$  ( $\text{C}_6\text{D}_6$ , 235.32 MHz)  $\delta$ :

$-57.7$  (s, 12F, *o*- $\text{CF}_3$ ),  $-63.1$  (s, 6F, *p*- $\text{CF}_3$ ) ppm.  $\text{C}_{18}\text{H}_4\text{Cl}_2\text{F}_{18}\text{Sn}$  (751.8): calculated: C, 28.8%; H, 0.5%; Cl, 9.4%; Sn, 15.8%; found: C, 27.8%; H, 1.0%; Cl, 9.2%; Sn, 14.0%.

### 3.5. Bis[2,4,6-tris(trifluoromethyl)phenyl]tin(IV) dibenzoate (5)

Dry acetonitrile (20 ml) was added to a mixture of 1.35 g (1.8 mmol) of **4** and 0.82 g (3.6 mmol) of  $\text{AgO}_2\text{CC}_6\text{H}_5$  and stirred for 24 h at room temperature with the exclusion of light. The microcrystalline white precipitate ( $\text{AgCl}$ ) was removed by filtration and all volatiles were evaporated in vacuo. The sticky residue was washed with 5 ml of hexane and recrystallized from 15 ml of hexane ( $-78^\circ\text{C}$ ). After drying under vacuum, 0.65 g (0.7 mmol, 39%) of the colourless, microcrystalline product was isolated; m.p.  $129^\circ\text{C}$ .

IR ( $\text{cm}^{-1}$ ): 3063 w (C–H arom.), 1649 m, 1627 m, 1603 m, 1582 m, 1493 m ( $\text{CF}_3$ ), 1336 m, 1307 s, 1281 s, 1261 s, 1195 vs br, 1136 vs br, 1120 vs, 1986 s, 1069 m sh, 912 s, 857 s ( $\text{R}_\text{F}$  out-of-plane), 802 m br, 724 vs, 685 vs, 668 m, 590 w, 560 w, 460 m, 436 m, 404 m, 366 s. EI-MS  $m/z$  (%): 801 ( $\text{M}^+ - \text{PhCOO}$ , 18), 643 ( $(\text{R}_\text{F})_2\text{Sn}^+ - 2\text{HF}$ , 16), 241 ( $\text{R}_\text{F}^+ - 2\text{HF}$ , 14), 77 ( $\text{Ph}^+$ , 20).  $^1\text{H NMR}$  ( $\text{C}_6\text{D}_6$ , 250 MHz)  $\delta$ : 7.74–7.61 (m, 10H,  $\text{C}_6\text{H}_5$ ), 7.55 (s, 4H,  $\text{C}_6\text{H}_2$ ) ppm.  $^{19}\text{F NMR}$  ( $\text{C}_6\text{D}_6$ , 235.32 MHz)  $\delta$ :  $-56.4$  (s,  $^4J(^{19}\text{F}-^{117/119}\text{Sn}) = 5.1$  Hz, 12F, *o*- $\text{CF}_3$ ),  $-63.6$  (s, 6F, *p*- $\text{CF}_3$ ) ppm.  $\text{C}_{32}\text{H}_{14}\text{F}_{18}\text{O}_4\text{Sn}$  (923.1): calculated: C, 41.6%; H, 1.5%; Sn, 12.9%; found: C, 40.3%; H, 1.8%; Sn, 14.1%.

### 3.6. Bis[2,4,6-tris(trifluoromethyl)phenyl]tin(IV)-bis(*o*-fluorobenzoate) (6)

A mixture of 1.78 g (6.0 mmol) of  $\text{AgO}_2\text{CC}_6\text{H}_4\text{F-}o$  and 2.26 g (3.0 mmol) of **4** in 50 ml of dry acetonitrile was stirred for 4 h in the dark. The reaction mixture was then filtered through Celite and the filtrate was taken to dryness; 1.91 g (2.0 mmol, 66%) of the product was isolated as a white, microcrystalline solid; m.p.  $140^\circ\text{C}$ .

IR ( $\text{cm}^{-1}$ ): 3096 w, 3069 w sh, 3039 w sh, 1702 m sh, 1617 s, 1599 m, 1587 m sh, 1546 m, 1496 m, 1466 m, 1460 m sh, 1420 m, 1386 m, 1298 vs, 1282 vs, 1272 s sh, 1262 m sh, 1235 m, 1198 vs, 1161 vs, 1150 vs, 1140 vs, 1131 vs, 1102 m, 1087 m, 916 m, 899 m, 857 m, 837 w, 825 w, 755 m, 694 m, 687 m, 670 m, 634 w, 610 w, 558 w, 521 w, 454 w, 434 w, 410 w, 367 w, 277 w, 224 w. EI-MS  $m/z$  (%): 821 ( $\text{M}^+ - \text{O}_2\text{CC}_6\text{H}_4\text{F}$ , 16), 679 ( $(\text{R}_\text{F})_2\text{Sn}^+$ , 18), 399 ( $\text{Sn}(\text{O}_2\text{CC}_6\text{H}_4\text{F})_2^+$ , 3), 263 ( $\text{R}_\text{F}\text{H}^+ - \text{F}$ , 2), 259 ( $\text{SnO}_2\text{CC}_6\text{H}_4\text{F}^+$ , 8), 243 ( $\text{R}_\text{F}^+ - 2\text{F}$ , 10), 123 ( $\text{FC}_6\text{H}_4\text{CO}^+$ , 100), 95 ( $\text{FC}_6\text{H}_4^+$ , 6), 75 ( $\text{C}_6\text{H}_3^+$ , 27).  $^1\text{H NMR}$  ( $\text{C}_6\text{D}_6$ , 250 MHz)  $\delta$ : 8.16 (s, 2H,  $\text{C}_6\text{H}_4\text{F}$ ), 8.12 (s, 2H,  $\text{C}_6\text{H}_4\text{F}$ ), 7.88 (s, 4H,  $\text{C}_6\text{H}_2$ ), 7.55 (s, 4H,  $\text{C}_6\text{H}_4\text{F}$ ) ppm.  $^{19}\text{F NMR}$  ( $\text{C}_6\text{D}_6$ , 235.32 MHz)  $\delta$ :  $-57.1$  (s, 2F,  $\text{C}_6\text{H}_4\text{F}$ ),  $-57.8$  (s, 12F, *o*- $\text{CF}_3$ ),  $-63.4$  (s, 6F, *p*- $\text{CF}_3$ ) ppm.  $\text{C}_{32}\text{H}_{12}\text{F}_{20}\text{O}_4\text{Sn}$  (959.1): calculated: C, 40.1%; H, 1.3%; Sn, 12.4%; found: C, 40.4%; H, 1.3%; Sn, 13.2%.

### 3.7. Bis(mesitylthio)bis[2,4,6-tris(trifluoromethyl)phenyl]tin(IV) (7)

A mixture of 1.36 g (2.0 mmol) of **1** and 0.61 g (2.0 mmol) of dimesityldisulphide in 50 ml of hexane was stirred at room temperature. After 2 days, the product had formed as a precipitate. Filtration and drying in vacuo afforded 1.13 g (1.1 mmol, 57%) of pale yellow **7**; m.p. 156 °C.

EI-MS  $m/z$  (%): 984 ( $M^+$ , 1), 965 ( $M^+ - F$ , 1), 867 ( $(R_F)_2Sn(SH)_2^+$ , 1), 833 ( $M^+ - SMes$ , 100), 683 ( $(R_F)_2Sn^+$ , 46), 545 ( $(R_F)_2Sn^+ - 2CF_3$ , 4), 401 ( $R_FSn^+$ , 12), 271 ( $SnSMes^+$ , 16), 243 ( $R_F^+ - 2HF$ , 56), 151 ( $SMes^+$ , 28), 119 ( $Mes^+$ , 18), 69 ( $CF_3^+$ , 8).  $^1H$  NMR ( $C_6D_6$ , 250 MHz)  $\delta$ : 7.91 (s,  $^4J(^1H-^{117/119}Sn) = 7.5$  Hz, 4H,  $R_F-H$ ), 6.59 (s, 4H,  $Mes-H$ ), 2.31 (br,  $\nu_{1/2} = 17$  Hz, 12H,  $o-CH_3$ ), 2.29 (s,  $^7J(^1H-^{117/119}Sn) = 8.4$  Hz, 6H,  $p-CH_3$ ) ppm.  $^{19}F$  NMR ( $C_6D_6$ , 235.32 MHz)  $\delta$ : -55.0 (br,  $\nu_{1/2} = 139$  Hz, 6F,  $o-CF_3$ ), -58.8 (br,  $\nu_{1/2} = 121$  Hz, 6F,  $o-CF_3$ ), -64.0 (s, 6F,  $p-CF_3$ ) ppm.  $^{13}C$  NMR ( $CDCl_3$ , 62.89 MHz)  $\delta$ : 144.4 (s,  $R_F-ipso-C$ ), 143.3 (s,  $Mes-ipso-C$ ), 137.9 (s,  $^5J(^{13}C-^{117/119}Sn) = 12.1$  Hz,  $Mes-p-C$ ), 136.9 (q,  $^2J(^{13}C-^{19}F) = 31.3$  Hz,  $F_3C-C$ ), 132.5 (q,  $^2J(^{13}C-^{19}F) = 34.7$  Hz,  $F_3C-C$ ), 128.5 (s,  $^4J(^{13}C-^{117/119}Sn) = 10.2$  Hz,  $Mes-m-C$ ), 127.1 (s br,  $\nu_{1/2} = 17$  Hz,  $R_F-m-C$ ), 126.5 (s br,  $\nu_{1/2} = 20$  Hz,  $Mes-o-C$ ), 123.0 (q,  $^1J(^{13}C-^{19}F) = 275.6$  Hz,  $CF_3$ ), 122.4 (q,  $^1J(^{13}C-^{19}F) = 272.0$  Hz,  $CF_3$ ), 22.6 (s,  $^6J(^{13}C-^{117/119}Sn) = 4.8$  Hz,  $Mes-p-CH_3$ ), 20.5 (s,  $^4J(^{13}C-^{117/119}Sn) = 4.4$  Hz, double intensity,  $Mes-o-CH_3$ ) ppm.  $C_{36}H_{26}F_{18}S_2Sn$  (983.4): calculated: C, 44.0%; H, 2.7%; Sn, 12.1%; found: C, 41.9%; H, 2.4%; Sn, 12.6%.

### 3.8. Bis[2,4,6-tris(trifluoromethyl)phenyl]-bis[2,4,6-tris(trifluoromethyl)phenylseleno]tin(IV) (8)

A mixture of 1.36 g (2.0 mmol) of **1** and 1.43 g (2.0 mmol) of bis[2,4,6-tris(trifluoromethyl)phenyl]diselenide was stirred in 50 ml of hexane at room temperature. After 2 h, a bright orange–yellow precipitate had formed, which was separated by filtration and dried in vacuo to afford 1.79 g (1.3 mmol, 64%) of **8**; m.p. 109 °C.

EI-MS  $m/z$  (%): 722 ( $(R_F)_2SnSe^+ - 2HF$ , 42), 703 ( $(R_F)_2SnSe^+ - 3F - 2H$ , 10), 670 ( $(R_F)_2Sn^+$ , 1), 642 ( $(R_F)_2Sn^+ - 2HF$ , 6), 415 ( $(R_F)_2Se^+ - F - 3C$ , 40), 361 ( $R_FSeH^+$ , 100), 342 ( $R_FSe^+ - F$ , 56), 321 ( $R_FSe^+ - H - 2F$ , 22), 282 ( $R_FH^+$ , 36), 263 ( $R_FH^+ - F$ ,

38), 241 ( $R_F^+ - 2HF$ , 30), 69 ( $CF_3^+$ , 34), 43 ( $C_2F^+$ , 36).  $^1H$  NMR ( $C_6D_6$ , 250 MHz)  $\delta$ : 7.72 (s,  $^5J(^1H-^{117/119}Sn) = 9$  Hz, 4H,  $R_F-H$ ), 7.66 (s, 4H,  $SeR_F-H$ ) ppm.  $^{19}F$  NMR ( $C_6D_6$ , 235.32 MHz)  $\delta$ : -56.4 (s br,  $\nu_{1/2} = 14$  Hz, 12F,  $SeR_F-o-CF_3$ ), -57.9 (s br,  $\nu_{1/2} = 14$  Hz, 12F,  $R_F-o-CF_3$ ), -63.6 (s,  $^6J(^{19}F-^{117/119}Sn) = 10.9$  Hz, 6F,  $R_F-p-CF_3$ ), -63.7 (s, 6F,  $SeR_F-p-CF_3$ ) ppm.  $^{77}Se$  NMR ( $THF-C_6D_6$ , 47.70 MHz)  $\delta$ : 255.3 (s br,  $\nu_{1/2} = 168$  Hz) ppm.  $^{119}Sn$  NMR ( $THF-C_6D_6$ , 93.27 MHz)  $\delta$ : -224.1 (s br,  $\nu_{1/2} = 72$  Hz) ppm.  $C_{36}H_8F_{36}Se_2Sn$  (1403.7): calculated: C, 30.8%; H, 0.6%; Sn, 8.5%; found: C, 29.9%; H, 1.0%; Sn, 8.0%.

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