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Chemistry of the stable diarylstannylene $(R_F)_2Sn$ ($R_F \equiv$ 2,4,6-tris(trifluoromethyl)phenyl): cycloaddition reactions with heterocumulenes, 3,5-di-*t*-butyl-*o*-benzoquinone and S_4N_4 [1]

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Abstract

The stable diarylstannylene $(R_F)_2Sn$ (**1**) ($R_F \equiv$ 2,4,6-tris(trifluoromethyl)phenyl) readily undergoes cycloaddition reactions. Three-membered stannacycles are obtained with various heterocumulenes such as ketenes, keteneimines and thioketenes. A four-membered SnNSN ring system (**5**) is obtained when **1** is allowed to react with $Me_3SiN{=}S{=}NSiMe_3$. Five-membered inorganic heterocycles are isolated from cycloaddition reactions of **1** with 3,5-di-*t*-butyl-*o*-benzoquinone and S_4N_4. The new compounds have been fully characterized by elemental analyses and spectroscopic methods. © 1997 Elsevier Science S.A.

Keywords: Diarylstannylenes; Heterocumulenes; Nonafluoromesityl derivatives; Cycloaddition reactions; Tetrasulfur tetranitride

1. Introduction

During the past 10 years the 2,4,6-tris(trifluoromethyl)-phenyl substituent ('nonafluoromesityl', abbreviated as R_F) has been shown to be a highly versatile building block in main group chemistry [1–3]. Owing to its ideal combination of sterically and electronically stabilizing effects, this ligand has been successfully employed in the stabilization of low coordination numbers around various main-group elements such as phosphorus [2,4–9], tin [10–15], lead [16], indium [17], or thallium [18]. Of special interest are the readily accessible and unusually stable nonafluoromesityl derivatives of divalent tin and lead [3,12]. $(R_F)_2Pb$ was the first diarylplumbylene made [16]. However, its derivative chemistry is thus far limited to very few reactions involving replacement of the organic ligands. For example, a protolytic reaction with two equivalents of the acidic thiol R_FSH leads to elimination of nonafluoromesitylene and formation of the lead(II) thiolate $Pb(SR_F)_2$ [16]. Much more interesting and diverse is the chemistry of the stable diarylstannylene derivative $(R_F)_2Sn$ (**1**) [10–15]. In this contribution we report cycloaddition reactions of **1** leading to three-, four-, and five-membered ring systems containing tin.

2. Results and discussion

In contrast to its lead(II) homolog $(R_F)_2Pb$, the stable stannylene derivative bis[2,4,6-tris(trifluoromethyl)-phenyl]tin $(R_F)_2Sn$ (**1**) readily undergoes cycloaddition reactions to afford the corresponding tin(IV) species [11]. We have studied reactions of **1** with various heterocumulenes, including ketenes, keteneimines and thioketenes, as well as with *N*,*N'*-bis(trimethylsilyl)sulfurdiimide, 3,5-di-*t*-butyl-*o*-benzoquinone, and tetrasulfur tetranitride. These reactions led to the formation of three-, four-, and five-membered ring systems containing tin. The results are summarized in Scheme 1.

A variety of cycloaddition reactions of stable or in situ generated stannylenes which afford three-membered heterocycles have been reported in the literature [19,20]. They include for example reactions of R_2M [21–32] or $R_2M{=}MR_2$ [13,33–42] (R = alkyl, aryl; $M \equiv$ Si, Ge, Sn) with unsaturated substrates to form three- and four-membered heterocycles. Ando and Tsumaraya reported the first reactions of germylenes with thioketenes [43]. The first stable three-membered methylenethiastannirane system derived from a

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[1] Dedicated to Professor Oskar Glemser on the occasion of his 85th birthday.

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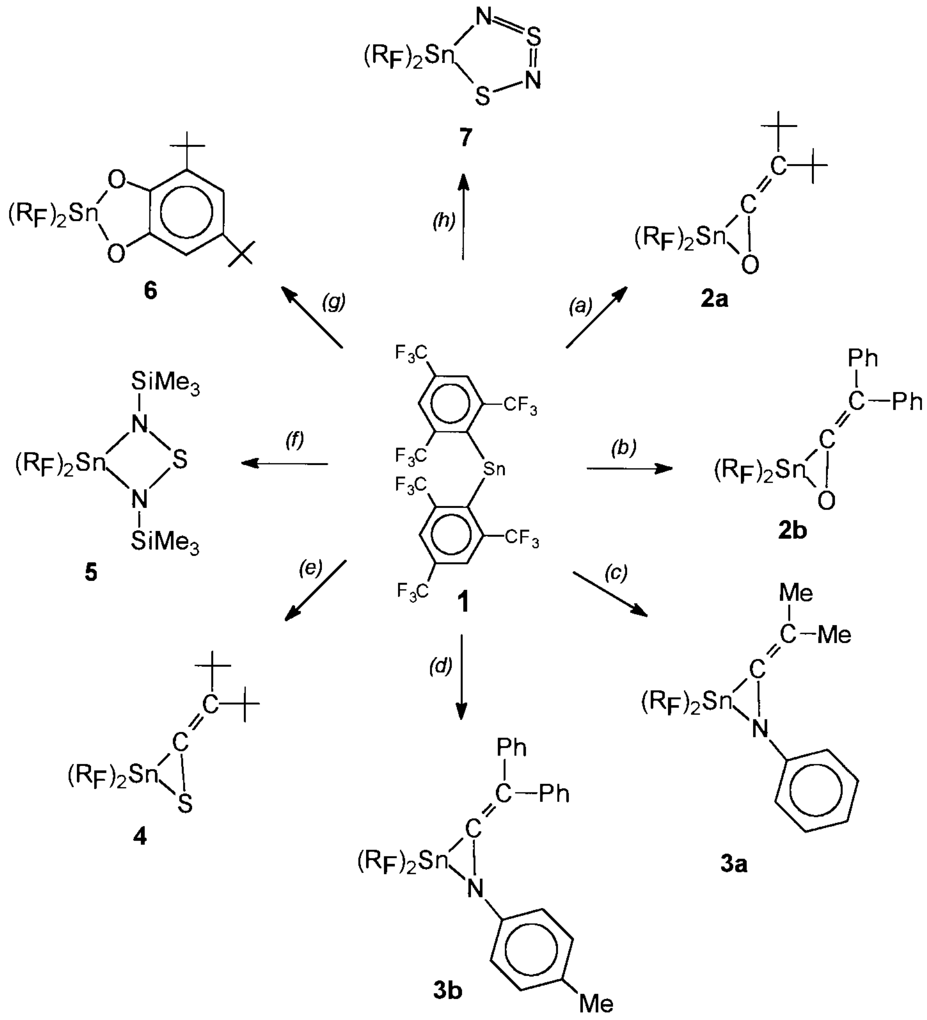

Scheme 1. Reactions of $(R_F)_2Sn$ (**1**) ($R_F \equiv 2,4,6\text{-}(CF_3)_3C_6H_2$). Reagents (a) *t*-$Bu_2C{=}C{=}O$, (b) $Ph_2C{=}C{=}O$, (c) $Me_2C{=}C{=}NPh$, (d) $Ph_2C{=}C{=}NC_6H_4Me$-*p*, (e) *t*-$Bu_2C{=}C{=}S$, (f) $Me_3SiN{=}S{=}NSiMe_3$, (g) 3,5-di-*t*-butyl-*o*-benzoquinone, (h) S_4N_4.

stannylene and a thioketene was prepared by Ohtaki et al. in 1993 [44]. Tin-containing three-membered rings are formed on treatment of **1** with different heterocumulenes such as ketenes, keteneimines, and thioketenes (Scheme 1). The reactions were carried out by prolonged heating (48 h) of the starting materials in THF solution. Subsequent crystallization of the crude products from hexane at −25 °C afforded the pure materials as almost colorless, pale yellow crystalline solids. The products **2**–**4** are readily soluble in common organic solvents such as pentane, hexane, toluene, diethylether, and THF. The compounds were characterized by elemental analyses and the usual combination of spectroscopic methods. All IR spectra of the compounds **2**–**4** show a set of bands characteristic of the nonafluoromesityl substituent (e.g. for **2b**: 1650, 1340, 1275, 1204, 1174 and 914 cm^{-1}). The ^1H NMR spectra are quite simple and show one singlet for the nonafluoromesityl aromatic ring protons besides the resonances resulting from the ‘heterocumulene part’ of the molecules. The ^{19}F NMR spectra exhibit only two singlets for the *ortho* and *para* CF_3 groups of the nonafluoromesityl substituents. In the cases of the ketene adducts **2a** and **2b** the molecular ion is detected with low intensity in the EI mass spectra. The other spectra show characteristic fragments of the product molecules and the nonafluoromesityl ligands.

A four-membered ring system derived from $(R_F)_2Sn$ was obtained when **1** was allowed to react with *N*,*N*′-bis(trimethylsilyl)sulfurdiimide in boiling THF. The crude product was isolated as a red oil which could be recovered in the form of orange–red crystals by crystallization from hexane. Characteristic IR absorption bands for **5** appear at 1261, 1195, and 1152 cm^{-1}. In the ^1H NMR spectrum the resonance of the aromatic nonafluoromesityl protons is observed at δ 7.96 ppm. The chemical equivalence of the $SiMe_3$ groups in solution is underlined by the appearance of a singlet at δ 0.09 ppm. The ^{19}F NMR spectrum of **5** shows five singlets in a narrow range at δ −57.2, −57.4, −57.9, −58.2 and −63.6 ppm, indicating the presence of *cis*/*trans* isomers. The combined intensities of the four *o*-CF_3 resonances and the *p*-CF_3 resonance are in the expected ratio of 2:1. Similar observations had been made before for the reaction products of the dialkylstannylene $Sn[CH(SiMe_3)_2]_2$ with enones [45]. It should also be mentioned that a tin-containing sulfur–nitrogen ring system derived from a sulfur diimide precursor is known from the literature: treatment of Li_2N_2S with equimolar amounts of *t*-Bu_2SnCl_2 afforded *t*-$Bu_2Sn(N{=}S{=}N)_2Sn$*t*-Bu_2, which contains an eight-membered $Sn_2S_2N_4$ ring system [46].

Two reactions have been studied in which the $(R_F)_2Sn$ unit becomes part of a five-membered heterocycle. This is the case when $(R_F)_2Sn$ is treated with either 3,5-di-*t*-butyl-*o*-benzoquinone or S_4N_4. Cycloaddition reactions of stannylenes leading to five-membered ring systems are known from the literature. For example, dialkoxystannylenes have been shown to add benzil to afford the corresponding five-membered cycloaddition products [47]. Stannadioxolenes are also formed during the reaction of photochemically or thermally generated dialkylstannylenes with 1,2-diketones [48]. Similarly, stannylenes react with vinyl ketones with formation of oxa-2-stannacyclopent-4-ene ring systems [49,50]. In this context, 3,5-di-*t*-butyl-*o*-benzoquinone is another useful reagent for reactions of this type. Prolonged heating of a mixture of **1** and 3,5-di-*t*-butyl-*o*-benzoquinone in boiling THF afforded the cycloaddition product **6** as a light yellow solid, which was characterized by elemental analysis as well as IR, ^1H NMR, and ^{19}F NMR spectra. A second tin–sulfur–nitrogen ring system (cf. **5**) was prepared by treatment of **1** with S_4N_4. The reaction was carried out in the usual manner by refluxing the starting materials for 48 h in THF solution. Bright yellow crystals of **7** (m.p. 183 °C) were isolated by recrystallization of the crude product from hexane. IR bands typical of the nonafluoromesityl units are found at 1624, 1295, 1264, 1200, 1116, and 915 cm^{-1}. The ^1H NMR spectrum of **7** exhibits a single resonance for the aromatic nonafluoromesityl protons at δ 7.63 ppm with a $^3J(^{117/119}Sn\text{-}^1H)$ coupling constant of 18 Hz. The EI mass spectrum shows the molecular ion at m/z 774 with 100% relative intensity as well as characteristic fragment peaks of the R_F substituents. The isotopic pattern of the molecular ion peak is identical with that calculated for $C_{18}H_4F_{18}N_2S_2$. Compound **7** belongs to a group of five-membered SnS_2N_2 rings which were explored many years ago by Roesky and coworkers [51]. For example, $Me_2SnS_2N_2$ was prepared by treatment of S_4N_4 with $N(SnMe_3)_3$. The direct reaction of a stannylene with tetrasulfur tetranitride described here represents a new access to this class of inorganic heterocycles [52].

The present investigation has demonstrated that the stable diarylstannylene $(R_F)_2Sn$ (**1**) is a useful precursor for cycloaddition reactions leading to three-, four-, and five-membered tin-containing ring systems. Some of the products might be of interest as starting materials for further reactions such as the complexation of transition metals or the formation of tin-containing heterocumulenes via desulfurization or deoxygenation reactions.

3. Experimental part

All reactions involving the stannylene **1** were carried out under dry, purified nitrogen. Solvents were dried over Na/benzophenone and were freshly distilled under N_2 prior to use. IR spectra: Bio-Rad FTS 7 (Nujol/KBr). 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 [2], R_FLi [1], $(R_F)_2Sn$ (**1**) [11], *t*-$Bu_2C{=}C{=}O$ [53], $Ph_2C{=}C{=}O$ [54], $Me_2C{=}C{=}NPh$ [55], $Ph_2C{=}C{=}NC_6H_4Me$-*p* [56,57], *t*-$Bu_2C{=}C{=}S$ [58], $Me_3SiN{=}S{=}NSiMe_3$ [59], and S_4N_4 [60] were prepared according to literature procedures. 3,5-Di-*t*-butyl-*o*-benzoquinone was purchased and used as received.

3.1. Cycloaddition reactions of $(R_f)_2Sn$ (1) (general procedure)

2.78 g (4.0 mmol) **1** and 4.0 mmol of the substrate are dissolved in 80 ml of THF and the mixture is stirred at reflux temperature for 48 h. The resulting solution is filtered through a thin layer of Celite filter aid and the filtrate is evaporated to dryness. The residue is extracted with 50 ml of boiling hexane and filtered again, using Celite. Concentration of the filtrate to a total volume of 10–20 ml and cooling to −25 °C (24 h) affords the crystalline product.

2a. Using 0.62 g *t*-$Bu_2C{=}C{=}O$, 0.97 g (29%) of **2a** are isolated as pale yellow crystals. M.p. 204 °C. Found: C, 39.0; H, 2.2. $C_{28}H_{22}F_{18}OSn$ (835.1) calculated: C, 40.3; H, 2.7%. IR (cm^{-1}): 1627 w, 1299 vs, 1264 s, 1195 s, 1154 s, 1137 vs, 915 s, 856 s, 815 vs. EI-MS m/z (%) 836 (M^+, 2), 779 (M^+-C_4H_9, 18), 243 (R_F^+-2F, 24), 57 ($C_4H_9^+$, 100). ^1H NMR (C_6D_6, 250 MHz) δ: 7.94 (s, 4 H, $^3J(^{117/119}Sn\text{-}^1H) = 23$ Hz, C_6H_2), 1.44 (s, 9 H, $C(CH_3)_3$) ppm. ^{19}F NMR (C_6D_6, 235.32 MHz) δ: −58.1 (s, 12 F, *o*-CF_3), −63.5 (s, 6 F, *p*-CF_3) ppm.

2b. From 0.78 g $Ph_2C{=}C{=}O$, 1.14 g (39%) of **2b** are obtained as a pale yellow solid. M.p. 253 °C. Found: C, 42.5; H, 1.3. $C_{32}H_{14}F_{18}OSn$ (875.1) calculated: C, 43.9; H, 1.6%. IR (cm^{-1}): 1740 s, 1650 s, 1340 s, 1307 vs, 1275 s, 1204 s, 1174 s, 953 vs, 914 s, 897 s, 847 s, 762 w, 743 s, 679 w. EI-MS m/z (%) 876 (M^+, 3), 243 (R_F^+-2F, 50), 194 (Ph_2CCO^+, 60), 165 (Ph_2C^+, 100). ^1H NMR (C_6D_6, 250 MHz) δ: 7.72 (s, 4 H, C_6H_2), 7.05 (m, 4 H, *o*-C_6H_5), 7.02 (m, 4 H, *m*-C_6H_5), 6.94 (m, 2 H, *p*-C_6H_5) ppm. ^{19}F NMR (C_6D_6, 235.32 MHz) δ: −58.1 (s, 12 F, *o*-CF_3), −63.3 (s, 6 F, *p*-CF_3) ppm.

3a. 0.58 g $Me_2C{=}C{=}NPh$ afford 1.34 g (39%) of **3a** as a light yellow solid. M.p. 225 °C. Found: C, 39.2; H, 2.0; N, 2.0. $C_{28}H_{15}F_{18}NSn$ (826.1) calculated: C, 40.7; H, 1.8; N, 1.7%. IR (cm^{-1}): 1998 s, 1734 s, 1662 vs, 1626 s, 1599 vs, 1505 vs, 1280 s, 1202 s, 1153 s, 914 s, 856 s, 819 s, 800 s, 761 s, 733 vs, 691 s, 671 s. EI-MS m/z (%) 243 (R_F^+-2F, 90), 145 (Me_2CCNPh^+, 80), 77 ($C_6H_5^+$, 22). ^1H NMR (C_6D_6, 250 MHz) δ: 7.94 (s, 4 H, $^3J(^{117/119}Sn\text{-}^1H) = 24$ Hz, C_6H_2), 7.71 (s, 2 H, *o*-C_6H_5), 7.53 (d, 1 H, *m*-C_6H_5), 7.31 (dd, 1 H, *p*-C_6H_5), 1.44 (s, 6 H, CH_3) ppm. ^{19}F NMR (C_6D_6, 235.32 MHz) δ: −58.1 (s, 12 F, $^4J(^{117/119}Sn\text{-}^{19}F) = 51$ Hz, *o*-CF_3), −63.2 (s, 6 F, *p*-CF_3) ppm.

3b. The reaction with 0.64 g $Ph_2C{=}C{=}NC_6H_4Me$-*p* yields 0.90 g (23%) of **3b** as a pale yellow solid. M.p. 264 °C. Found: C, 47.0; H, 1.9; N, 1.3. $C_{39}H_{21}F_{18}NSn$ (964.3) calculated: C, 48.6; H, 2.2; N, 1.5%. IR (cm^{-1}): 1998 vs, 1734 s, 1661 s, 1599 s, 1580 s, 1503 s, 1493 m, 1280 m, 1200 s, 1154 s, 915 s, 865 s, 801 vs, 758 s, 733 m, 693 s. ^1H NMR (C_6D_6, 250 MHz) δ: 7.94 (s, 4 H, C_6H_2), 7.85 (s, 2 H, *o*-C_6H_4Me), 7.68 (m, 2 H, *m*-C_6H_4Me), 7.51 (m, 2 H, *p*-C_6H_5), 7.01–6.82 (m, 8 H, *o,m*-C_6H_5), 1.80 (s, 3 H, CH_3) ppm. ^{19}F NMR (C_6D_6, 235.32 MHz) δ: -56.4 (s, 6 F, $^4J(^{117/119}Sn\text{-}^{19}F) = 72$ Hz, *o*-CF_3), -57.2 (s, 6 F, $^4J(^{117/119}Sn\text{-}^{19}F) = 72$ Hz, *o*-CF_3), -63.5 (s, 6 F, *p*-CF_3) ppm.

4. From 0.68 g di-*t*-butylthioketene are isolated 1.36 g (40%) of **4** as an almost colorless solid. M.p. 212 °C. Found: C, 38.2; H, 2.3; S, 2.3. $C_{28}H_{22}F_{18}SSn$ (851.2) calculated: C, 39.5; H, 2.6; S, 3.8%. IR (cm^{-1}): 1734 s, 1627 vs, 1583 s, 1297 s, 1282 vs, 1236 vs, 1197 s, 1137 s, 914 s, 658 vs. EI-MS *m/z* (%) 676 (M^+-$2C_4H_9$, 50), 455 (M^+-R_F-$2C_4H_9$, 21), 243 (R_F^+-2F, 56), 57 ($C_4H_9^+$, 100). ^1H NMR (C_6D_6, 250 MHz) δ: 7.90 (s, 4 H, C_6H_2), 1.28 (s, 18 H, $C(CH_3)_3$) ppm. ^{19}F NMR (C_6D_6, 235.32 MHz) δ: -58.2 (s, 12 F, $^4J(^{117/119}Sn\text{-}^{19}F) = 94$ Hz, *o*-CF_3), -63.6 (s, 6 F, *p*-CF_3) ppm.

5. The reaction with 0.84 g bis(trimethylsilyl)sulfurdiimide affords 1.42 g (34%) of **5** as an orange–red solid. M.p. 185 °C. Found: C, 31.2; H, 2.7; N, 2.1. $C_{24}H_{22}F_{18}N_2SSi_2Sn$ (887.3) calculated: C, 32.5; H, 2.5; N, 3.2%. IR (cm^{-1}): 2350 w, 1261 vs, 1199 s, 1152 vs, 1089 s, 1018 vs, 914 s, 850 w, 803 vs, 721 s, 693 s, 667 w, 620 w. ^1H NMR (C_6D_6, 250 MHz) δ: 7.96 (s, 4 H, C_6H_2), 0.09 (s, 18 H, $SiMe_3$) ppm. ^{19}F NMR (C_6D_6, 235.32 MHz) δ: -57.2 (s, 3 F, $^4J(^{117/119}Sn\text{-}^{19}F) = 15$ Hz, *o*-CF_3), -57.4 (s, 3 F, $^4J(^{117/119}Sn\text{-}^{19}F) = 15$ Hz, *o*-CF_3), -58.0 (s, 3 F, $^4J(^{117/119}Sn\text{-}^{19}F) = 27$ Hz, *o*-CF_3), -58.2 (s, 3 F, $^4J(^{117/119}Sn\text{-}^{19}F) = 27$ Hz, *o*-CF_3), -63.6 (s, 6 F, *p*-CF_3) ppm.

6. 0.88 g 3,5-di-*t*-butyl-*o*-benzoquinone yield 0.67 g (17%) **6** as a pale yellow solid. M.p. 187 °C. Found: C, 41.8; H, 2.9. $C_{32}H_{24}F_{18}O_2Sn$ (901.2) calculated: C, 42.7; H, 2.7%. IR (cm^{-1}): 1625 s, 1578 s, 1337 s, 1297 s, 1280 vs, 1262 s, 1203 vs, 1153 vs, 913 vs, 854 vs. ^1H NMR (C_6D_6, 250 MHz) δ: 8.03 (s, 4 H, $^3J(^{117/119}Sn\text{-}^1H) = 24$ Hz, C_6H_2), 7.90 (s, 1 H, $C_6H_2O_2$), 6.90 (s, 1 H, $C_6H_2O_2$), 1.53 (s, 9 H, $C(CH_3)_3$), 1.24 (s, 9 H, $C(CH_3)_3$) ppm. ^{19}F NMR (C_6D_6, 235.32 MHz) δ: -58.9 (s, 12 F, $^4J(^{117/119}Sn\text{-}^{19}F) = 54$ Hz, *o*-CF_3), -63.6 (s, 6 F, *p*-CF_3) ppm.

7. The reaction with 0.74 g S_4N_4 affords 0.94 g (58%) **7** as a bright yellow solid. M.p. 181 °C. Found: C, 29.1; H, 1.4; N, 3.5; S, 7.5. $C_{18}H_4F_{18}N_2S_2Sn$ (773.0) calculated: C, 28.0; H, 0.5; N, 3.6; S, 8.3%. IR (cm^{-1}): 1624 vs, 1579 s, 1491 w, 1295 s, 1278 vs, 1264 s, 1200 s, 1116 vs, 915 s, 854 s, 798 s, 692 vs, 685 vs. EI-MS *m/z* (%) 774 (M^+, 100), 493 (M^+-R_F-F, 64), 415 (R_FSnN^+, 38), 401 (R_FSn^+, 96), 281 (R_F^+, 52), 243 (R_F^+-2F, 98). ^1H NMR (C_6D_6, 250 MHz) δ: 7.63 (s, 4 H, $^3J(^{117/119}Sn\text{-}^1H) = 8$ Hz, C_6H_2) ppm. ^{19}F NMR (C_6D_6, 235.32 MHz) δ: -57.2 (s, 12 F, $^4J(^{117/119}Sn\text{-}^{19}F) = 12$ Hz, *o*-CF_3), -63.5 (s, 6 F, $^4J(^{117/119}Sn\text{-}^{19}F) = 5$ Hz, *p*-CF_3) ppm.

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