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Oxidative Addition to a Monomeric Stannylene To Give Four-Coordinate Tin Compounds Containing the Bulky Bidentate Ligand $C(SiMe_3)_2SiMe_2CH_2CH_2Me_2Si(Me_3Si)_2C$. Crystal Structures of $\overline{CH_2Me_2Si(Me_3Si)_2CSnC(SiMe_3)_2SiMe_2CH_2}$, $\overline{CH_2Me_2Si(Me_3Si)_2CSnMe(OCOCF_3)C(SiMe_3)_2SiMe_2CH_2}$, and $(CF_3COO)_2MeSnC(SiMe_3)_2SiMe_2CH_2CH_2Me_2Si(Me_3Si)_2CSnMe(OCOCF_3)_2$

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Reaction of $(THF)_2KRRK(THF)_2$ ($RR = C(SiMe_3)_2SiMe_2CH_2CH_2Me_2Si(Me_3Si)_2C$) with $SnCl_2$ in Et_2O gave a mixture of the cyclic and linear Sn^{II} compounds $\overline{RSnR}$, **4**, and ClSnRRSnCl, **9**. This mixture was treated with MeI to give the corresponding Sn^{IV} compounds $\overline{RSnMeIR}$, **5**, and IClMeSnRRSnMeClI, **10**. Treatment of **5** and **10** with AgO_2CCF_3 gave the corresponding trifluoroacetates **6** and **11**, which were structurally characterized.

Introduction

During the last 15 years, we,[1] and more recently others,[2] have described numerous organometallic compounds containing the bulky “trisyl” ligand $C(SiMe_3)_3$ and have found them to show much interesting and unusual chemistry. Because of the size of the ligand, compounds in which there is only one $C(SiMe_3)_3$ substituent predominate; the only compounds containing two such substituents obtained until recently were the linear dialkyls of groups 2 and 12 and manganese and the isoelectronic ate complexes of groups 1 and 11.[1] Steric effects in these dialkyls account for their remarkable chemical stability compared with that of compounds bearing smaller alkyl groups; the zinc compound $Zn\{C(SiMe_3)_3\}_2$, for example, is stable in air and can be distilled in steam.[3] Later we isolated dialkyls of the larger metals Yb, Eu,[4] and Ca[5] and found that in each case the C−M−C skeleton was bent. We were thus led to ask whether it was possible to make three- or four-coordinate species containing two “trisyl” groups. We have not so far succeeded in isolating such species, but the development of the bidentate “trisiamyl” ligand, effectively two “trisyl” groups joined like Siamese twins through peripheral methyl groups,[6] is providing new insight into organometallic compounds having bonds from the metal to two highly sterically hindered triorganosilyl-substituted carbon centers.

We found that the precursor $HC(SiMe_3)_2SiMe_2CH_2CH_2Me_2Si(Me_3Si)_2CH$ (HRRH) could be readily metalated by methyllithium,[6] -potassium,[2] or -cesium.[7] With MeLi cyclic lithates, for example **1** or the corresponding TMEDA complex (TMEDA = tetramethylethylenediamine), were obtained, but MeK or MeCs gave linear bimetalated compounds such as **2**. The lithium compounds reacted with slurries of the dihalides MCl_2 (M = Zn,[2] Hg,[6] or Pb[10]) to give chelated dialkyls $\overline{RMR}$, but with the four-coordinate $SnCl_4$ only the linear bimetalated species $Cl_3SnRRSnCl_3$ **3** was obtained irrespective of the alkali metal/tin mole ratio.[2] This was not surprising since the formation of a metallacycle containing four-coordinate tin would require reaction through a five-coordinate transition state between the sterically hindered organoalkali metal center in an intermediate such as $LiRRSnCl_3$ and the sterically hindered tin center in the same molecule. It seemed to us that if Sn^{IV} metallacycles were to be made, the ring would probably have to be formed from an Sn^{II} rather than an Sn^{IV} precursor, and the Sn^{IV} derivatives obtained subse-

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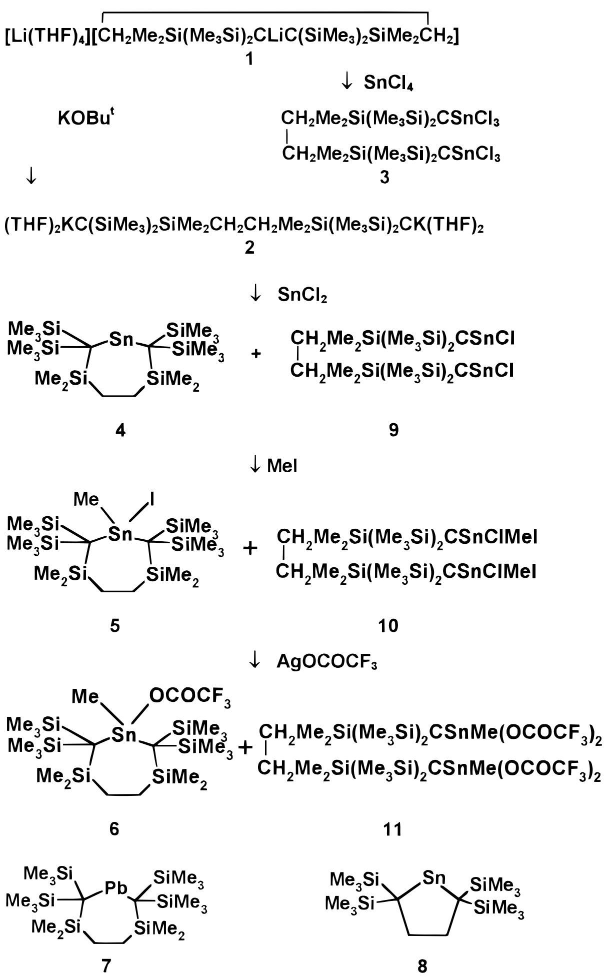
Scheme 1



quently by oxidative addition, a known route for the synthesis of a range of Sn^{IV} compounds from the less crowded $Sn\{CH(SiMe_3)_2\}_2$.[8,9] In this paper we show that this approach is successful, having isolated the Sn^{II} metallacycle **4** and converted it into the Sn^{IV} compound **5** by oxidation with iodomethane. The multinuclear NMR spectra of **5** were consistent with those of a metallacycle containing four-coordinate tin, and the incorporation of Sn into the ring was confirmed by the conversion of **5** into the trifluoroacetate **6**, which was structurally characterized. The formation of an open chain Sn^{II} species **9** analogous to the Sn^{IV} species **3** was shown by its conversion with $AgOCOCF_3$ into the tetratrifluoroacetate **11** (Scheme 1).

Results and Discussion

We indicated previously[10] that the reaction between the TMEDA complex analogous to **1** and $SnCl_2$ gave a red solution, which from NMR spectroscopic data appeared to contain the compound **4** as a TMEDA complex, but were unable to isolate crystals for an X-ray study. We have now found that the potassium compound **2** reacts with $SnCl_2$ in Et_2O to give a purple solution, from which crystals of the base-free compound **4**, isomorphous with the previously described lead analogue **7**,[10] could be obtained. Compound **4** was shown to be monomeric in the solid by a crystal structure determination, in the gas phase by the strong peak from the molecular ion in the mass spectrum, and in solution by the chemical shifts δ_{Sn} 2299 and δ_C (quaternary) 60.4, which are similar to those in monomeric $Sn\{CH(SiMe_3)_2\}_2$, δ_{Sn} 2328, δ_C 60.0,[11a] and the chelated Sn^{II} alkyl **8**, δ_{Sn} 2323, δ_C 69.5.[11b]

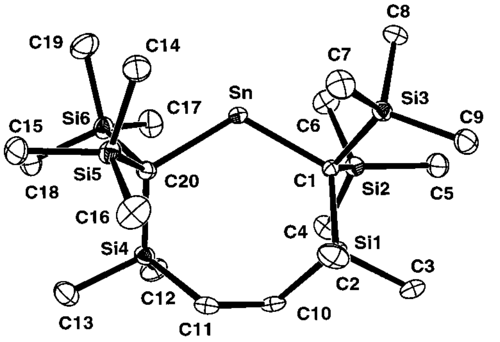


Figure 1. Molecular structure of $CH_2Me_2Si(Me_3Si)_2CSnC(SiMe_3)_2SiMe_2CH_2$, **4**.

The molecular structure of **4** and the most important bond lengths and angles are given in Table 1. The lattice consists of isolated molecules with no Sn−Sn interactions (Sn⋯Sn = 8.9 Å) such as those in $Sn\{CH(SiMe_3)_2\}_2$,[8] $Sn\{Si(SiMe_3)_3\}_2$,[12a] or the red form of $Sn\{C_6H_2(CF_3)_3\text{-}2,4,6\}_2$.[12b] There is no crystallographic symmetry in the molecule as a whole, but the central puckered ring has an approximate C_2 axis.

The bond distances within the bulky bidentate ligand in compounds **4** and **7** are the same within experimental error. There are small differences in metal−carbon distances, but these do not prevent the two molecules from packing in identical lattices. The C−Sn−C angle in **4** (118°) is larger than those in other Sn^{II} organometallic compounds, for example, **8** (87°),[11b] monomeric $Sn\{CH(SiMe_3)_3\}_2$ (96°),[13] or diaryl derivatives (97−115°),[14,15] but similar to the C−Pb−C angle in **7**,[10] suggesting that the angles in the trisiamyl compounds are particularly wide because of repulsion between the two trisyl-like halves of the chelated ligand. It is remarkable that the C−M−C angle in the metallacycle can vary from 166−170° in trisiamylzinc[2] or -mercury[6] (mean Zn−C 1.963, Hg−C 2.132 Å) to a value as low as 117−118° in **4** or **7** (mean Sn−C 2.285, Pb−C 2.404 Å). This is achieved by adjustments to M−C−Si angles, which in **4** and **7** range from 96° to 120°. The implication is that there is no

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Table 1. Selected Bond Lengths (Å) and Angles (deg) for Compounds 4, 6, and 11

	RSnR **4**	RSnMe(OCOCF$_3$)R **6**	(CF$_3$COO)$_2$(Me)SnR-RSn(Me)(OCOCF$_3$)$_2$ **11**
Sn−O		2.102(3)	2.046(6), 2.031(7)
Sn−C	2.284(3), 2.286(3)	2.227(5), 2.227(5)	2.119(7)
Sn−Me		2.160(4)	2.126(10)
C−Si	1.894(3)[a]	1.917(5)[a]	1.918(8)[a]
Si−Me	1.878(4)[a]	1.872(6)[a]	1.869(9)[a]
Si−CH$_2$	1.876(4)[a]	1.870(6)[a]	1.878(8)
C−C	1.535(5)	1.554(7)	1.548(16)
C−Sn−C	117.6(1)	132.75(19)	
O−Sn−Me		104.08(16)	108.0(4), 109.5(4)
O−Sn−C		97.81(17), 99.46(16)	108.9(3), 101.6(3)
O−Sn−O			92.2(3)
C−Sn−Me		108.38(18), 109.42(19)	130.0(4)
Sn−C−Si	120.1(2), 105.8(1), 97.6(1)	108.3(2)−112.2(2)	105.1(4), 105.3(3), 109.5(4)
C1−Si−CH$_2$	115.5(2), 115.6(2)	111.2(3), 115.3(2)	111.4(4)
Si−C−Si	110.7(2)[a]	105.1(2)−111.8(3)	112.4(4), 111.6(4), 112.4(4)
Me−Si−Me	103.6(2)−106.1(2)	102.8(3)−107.2(3)	104.6(5)−109.4(5)
C1−Si−Me	110.9(2)−115.3(2)	111.1(3)−116.6(2)	110.3(4)−114.2(4)
Si−C−C	118.2(2), 118.5(2)	116.3(4), 120.4(4)	112.4(8)
Me−Si−CH$_2$	103.8(2)−106.4(2)	103.7(3)−109.7(3)	109.4(4), 105.8(4)

[a] Average values with esd's of individiual measurements in parentheses. No value differs significantly from the mean.

steric reason compounds containing two C(SiMe$_3$)$_3$ groups at a four-coordinate metal center should not be stable. Their synthesis from four-coordinate precursors may however be difficult for kinetic reasons since there may not be room for two trisyl groups in a five-coordinate transition state.

The isolation of the Sn^{II} compound **4** containing a ligand with two bulky carbanionic centers made it possible for us to attempt syntheses of Sn^{IV} compounds by oxidative addition and thus avoid precursors involving crowded transition states. Compound **4** reacted rapidly with iodomethane to give a colorless air-stable solid that was identified as the oxidative addition product **5** by its C, H, and I analyses, by the presence in the mass spectrum of a peak from the molecular ion, and by multinuclear NMR data. The formation of a Sn^{IV} metallacycle, with iodine on one side and methyl on the other, was confirmed by the presence of two signals in each of the ^{1}H, ^{13}C, and ^{29}Si spectra assigned to SiMe$_3$ groups on either side of the ring and to signals from diastereotopic CH$_2$ protons. Since we were unable to obtain a sample suitable for an X-ray study, we decided to replace the iodide by another anionic ligand in the hope that the resulting derivative would give better crystals that would allow us to confirm that a Sn^{IV} metallacycle had indeed been formed.

We thus allowed a fresh sample of the potassium compound **2** to react with SnCl$_2$, added MeI to the resulting purple solution, removed solvent, and treated the white solid product with AgOCOCF$_3$ in CH$_2$Cl$_2$ in order to replace the iodide with trifluoroacetate. The solid silver iodide was separated, the organic product extracted with hexanes, and the solvent removed from the extract. An ^{19}F NMR spectrum of the residue showed the presence of two compounds in the ratio 33:67, and these were subsequently separated by fractional crystallization from hexanes. The NMR spectra of the first batch of colorless crystals showed single SiMe$_3$ and SiMe$_2$ peaks, and a structure determination confirmed that they consisted of the novel open chain derivative **11**. It is clear that in the synthesis of **4** there is competition from the reaction leading to the open chain Sn^{II} species **9**. In the subsequent reactions first with MeI and then with AgOCOCF$_3$, **9** is converted via **10** into the open chain tetratrifluoracetate **11**. A second crop of blocklike crystals was formed from the mother liquor, and these proved to be the desired cyclic trifluoroactetate **6**. It is likely that the proportions of **4** and **9** could be varied by changing the reaction conditions, e.g., the mol ratio **2**/SnCl$_2$, the solvent, or the concentration, but we have not so far studied this.

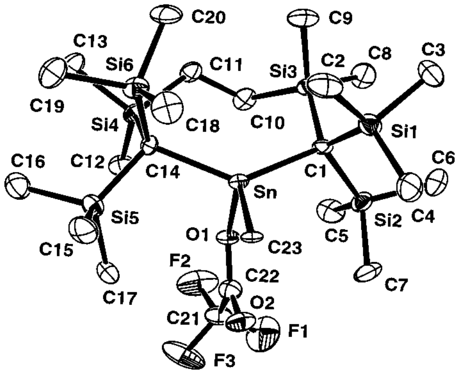

Figure 2. Molecular structure of CH$_2$Me$_2$Si(Me$_3$Si)$_2$CSnMe(OCOCF$_3$)C(SiMe$_3$)$_2$SiMe$_2$CH$_2$, **6**.

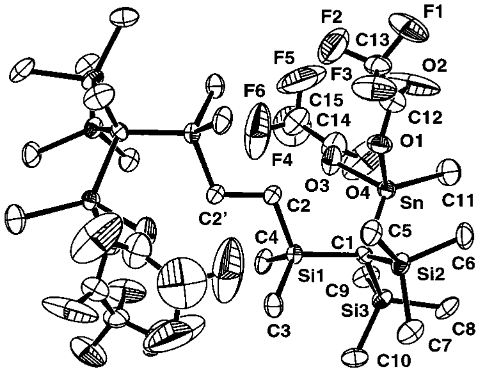

Figure 3. Molecular structure of (CF$_3$COO)$_2$MeSnC(SiMe$_3$)$_2$SiMe$_2$CH$_2$CH$_2$Me$_2$Si(Me$_3$Si)$_2$CSnMe(OCOCF$_3$)$_2$, **11**.

The molecular structures of compounds **6** and **11** are shown in Figures 2 and 3, and selected bond lengths and angles are given in Table 1. When the structure of the cyclic Sn^{II} compound **4** is compared with that of the

Sn^{IV} compound **6**, it is found that there are no significant differences between the bond lengths and angles within the bulky ligand. However, the Sn−C bonds are about 0.06 Å longer in the Sn^{II} than in the Sn^{IV} compound. This is consistent with a general trend; the bond lengths in similar sterically hindered [tris(organosilyl)methyl]tin(IV) compounds range from 2.120(11) to 2.185(2) Å,[16] whereas those in the two previously reported [tris(organosilyl)methyl]tin(II) compounds are 2.296(10) and 2.294(5) Å.[17] Similarly, the distances in $Sn\{CH(SiMe_3)_2\}_2HOH$ and gaseous $Sn\{CH(SiMe_3)_2\}_2$ are, respectively, 2.149(2)[9] and 2.24(1) Å.[13] (Bond lengths in Sn^{II}−aryl compounds are 2.21−2.23 Å.[14,15,18])

The C−Sn−C angle in the Sn^{IV} derivative **6** (133°)[19] is much wider than that in the Sn^{II} derivative **4** (118°), reflecting the relief of intraligand steric strain as the lone pair of electrons is converted into two extra bonds.[20] A similar expansion in C−Sn−C is found on going from monomeric SnR_2 (96°, R = $CH(SiMe_3)_2$)[13] to SnR_2HOH (115°)[9] or $R_2Sn(\mu\text{-}O)(\mu\text{-}O_2)SnR_2$ (124°),[21] or calculated for the transformation from $SnMe_2$ (99°) to $SnMe_3Cl$ (113°).[22]

The centrosymmetric structure of the bis(trifluoroacetate) **11** calls for little comment. The bond lengths and angles within the ligand are insignificantly different from those in **6**, and the configuration at tin, with wide C−Sn−C (130°) and narrow O−Sn−O (92°) angles, is similar to that in $\{[(Me_3Si)_3C]Me(O_2NO)Sn\}_2O$.[16b] The long Sn···O2 (3.087 Å) and Sn···O4 (3.142 Å) distances show that the carboxylate ligands are effectively monodentate.

Experimental Section

Air and moisture were excluded as far as possible by the use of flame-dried glassware and Ar blanket gas. NMR spectra were recorded at 300.13 (^1H), 125.8 (^{13}C), 150.84 (^{19}F), 99.4 (^{29}Si), and 186.6 MHz (^{119}Sn) from samples in C_6D_6, and chemical shifts are relative to $SiMe_4$ for H, C, and Si, $SnMe_4$ for Sn, and $CFCl_3$ for F; intensities of the quaternary and ^{29}Si signals were enhanced by polarization transfer. EI mass spectra were obtained at 70 eV; data are given for species containing ^{28}Si and ^{120}Sn. Compound **2** was synthesized as described previously.[2]

$\overline{\mathrm{CH_2Me_2Si(Me_3Si)_2CSnC(SiMe_3)_2SiMe_2CH_2}}$, 4. A solution of the potassium compound **2** (2.0 g, 2.4 mmol) in Et_2O (20 mL) was added dropwise to a stirred slurry of anhydrous $SnCl_2$ (0.45 g, 2.4 mmol) in Et_2O at −78 °C, and the mixture was allowed to warm slowly to room temperature then stirred for a further 3 h. The solvent was removed under vacuum and the purple residue extracted with hexane (30 mL). The volume of the extract was reduced to 10 mL and the solution kept at −20 °C to give purple air- and moisture-sensitive plates, 0.8 g (58%), mp 135 (softens 120 °C). Anal. Calcd for $C_{20}H_{52}Si_6Sn$: C, 41.4; H, 9.0. Found: C, 39.8; H. 9.1. UV/vis (hexane): λ_{max}/nm (ϵ/mol^{-1} L cm^{-1}) 546 (175), 356 (105). ^1H NMR: δ 0.27 (12H, s, $SiMe_2$), 0.32 (36H, s, $SiMe_3$), 1.10 (4H, s, CH_2). ^{13}C NMR: δ 5.4 ($^1J_{CSi}$ = 51 Hz, $^3J_{CSn}$ = ca. 40 Hz, $SiMe_3$), 6.8 ($^1J_{SiC}$ = 49 Hz, $SiMe_2$), 16.2 (CH_2), 60.4 ($^1J_{CSn}$ = ca. 540 Hz, $^1J_{CSi}$ = 32 Hz, CSi_3). ^{29}Si NMR: δ −10.1 ($^1J_{SiC}$ = 51 Hz, $^2J_{SiSn}$ 31 Hz, $SiMe_3$), −0.74 ($SiMe_2$). ^{119}Sn NMR: δ 2299. MS *m/z* 580 (10, M), 462 (5, M − Sn), 351 (18, $SnC(SiMe_3)_3$), 336 (35, $SnC(SiMe_3)_3$ − Me), 217 (100, $(Me_3Si)_2CHSiMe_2$). A crystal suitable for an X-ray study was obtained from hexane.

$\overline{\mathrm{CH_2Me_2Si(Me_3Si)_2CSnMeIC(SiMe_3)_2SiMe_2CH_2}}$, 5. Iodomethane (2.80 g, 8.0 mmol) was added to a solution of compound **4** (0.80 g. 3.0 mmol) in Et_2O (20 mL). The mixture became colorless after 20 min and was stirred for a further 2 h before the solvent was removed under reduced pressure, and the residue was crystallized from Et_2O/hexane to give a colorless solid, **5**, mp 86−88 °C. Yield: 2.5 g (65%). Anal. Calcd for $C_{21}H_{55}ISi_6Sn$: C, 34.95; H, 7.68; I, 17.58. Found: C, 34.35; H, 7.75; I, 17.67. ^1H NMR: δ 0.32 and 0.36 (6H, s, $SiMe_2$), 0.42 and 0.44 (18H, s, $SiMe_3$), 0.82−0.98 (4H, m, CH_2), 1.29 ($^2J_{HSn}$ = 42.5 Hz, 3H, s, SnMe). ^{13}C NMR: δ 5.49 and 5.53 ($SiMe_2$), 7.8 ($^1J_{SiC}$ = 52 Hz, $^3J_{CSn}$ = 19.0 Hz) and 8.1 ($^1J_{SiC}$ = 51.4 Hz, $^3J_{CSn}$ = 19.8 Hz, $SiMe_3$), 13.6 ($^3J_{CSn}$ = 19.1 Hz, CH_2), 15.6 ($^1J_{117SnC}$ = 257 Hz, $^1J_{119SnC}$ = 268 Hz, SnMe), 21.3 ($^1J_{SiC}$ = 57 and 28 Hz, $CSnSi_3$). ^{29}Si NMR: δ 0.4 ($^2J_{SiSn}$ = 44.7 Hz) and 0.7 ($^2J_{SiSn}$ = 41.7 Hz, $SiMe_3$), 2.2 ($^2J_{SiSn}$ = 24.3 Hz, $^1J_{SiC}$ = 57 Hz, $SiMe_2$). ^{119}Sn NMR: δ −42. MS: *m/z* 722 (2, M), 707 (20, M − Me), 595 (25, M − I), 477 (70, RSnI), 379 (80, RSnMe − CH_2), 363 (90), 218, (100, $(Me_3Si)_2(Me_2HSi)CH$), 201 (90, $(Me_3Si)(Me_2Si)_2C$).

$\overline{\mathrm{CH_2Me_2Si(Me_3Si)_2CSnMe(OCOCF_3)C(SiMe_3)_2SiMe_2CH_2}}$, 6, and $(CF_3COO)_2MeSnC(SiMe_3)_2SiMe_2CH_2CH_2Me_2Si(Me_3Si)_2CSnMe(OCOCF_3)_2$, 11

A sample of **2** (2.4 g) was treated with $SnCl_2$ in Et_2O as described in the preparation of **4** above, and the purple mixture was allowed to warm to room temperature and then filtered. An excess of MeI (3.0 g) was added to the filtrate and the solution stirred for 2 h. The solvent was then removed to leave a white solid residue. A sample of this (0.34 g), without further purification, was stirred with $AgOCOCF_3$ (0.81 g, 3.67 mmol) in CH_2Cl_2 (20 mL) at room temperature for 3 h. The mixture was then filtered, solvent removed from the filtrate under vacuum, and the residue extracted with hexane (3 × 10 mL). The extract was reduced to a volume of 10 mL and kept at 5 °C to give colorless plates of **11** (0.18 g), mp 138−140 °C. Anal. Calcd for $C_{30}H_{52}F_{12}O_8Si_6Sn_2$: C, 30.67; H, 4.47. Found: C, 30.52; H, 4.58. ^1H NMR: δ 0.14 (36H, s, $SiMe_3$), 0.29 (12H, s, $SiMe_2$) 0.8 (4H, s, CH_2), 1.26 (6H, s, $^2J_{SnH}$ = 65.6 Hz, SnMe).^{13}C NMR: δ 0.5 ($^1J_{SiC}$ = 52 Hz, $SiMe_2$), 4.5 ($^1J_{SiC}$ = 51 Hz, $SiMe_3$), 11.8 (CH_2), 13.1 (SnMe), 19.6 (CSi_3Sn) 115.9 (q, $^1J_{CF}$ = 287 Hz, CF_3), 159.9 (q, $^2J_{CF}$ = 41 Hz, CO). ^{29}Si NMR: δ −0.3 ($^1J_{SiC}$ = 52 Hz, $SiMe_3$), 3.5 ($^1J_{SnC}$ = 56 Hz, $SiMe_2$). ^{19}F NMR: δ −75.0. ^{119}Sn NMR: δ 1.9. IR: 1737 cm^{-1} (ν(CO)). MS: *m/z*:1167 (2, M − Me), 1069 (M − $COCF_3$), 577 (M/2 − Me), 201 $Me_3SiC(SiMe_2)_2$), 73 (100). A second crop of crystals was shown to be **6** (0.22 g), mp 171−173 °C. Anal. Calcd for $C_{23}H_{55}F_3O_2Si_6Sn$: C, 39.02; H, 7.83. Found: C, 39.05; H, 7.74. ^1H NMR: δ 0.22 and 0.36 (18H, s, $SiMe_3$), 0.28 and 0.29 (6H, s, $SiMe_2$), 0.8−1.09 (4H, m, CH_2), 1.32 (3H, s, SnMe). ^{13}C NMR: δ 3.5 (CH_2), 5.2 and 5.5 ($SiMe_2$), 6.9 and 7.7 ($SiMe_3$), 14.2 (SnMe), 23.8 ($CSnSi_3$), 116.0 (q, $^1J_{CF}$ = 291 Hz, CF_3), 158.5 ($^1J_{CF}$ = ca. 45 Hz, CO). ^{19}F NMR: δ −74.1. ^{29}Si NMR: δ −2.6 ($^2J_{SiSn}$ = 32

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Table 2. Summary of Crystallographic Data for 4, 6, and 11

	RSnR **4**	SnRRMeOCOCF$_3$ **6**	(CF$_3$COO)$_2$(Me)SnR-RSn(Me)(OCOCF$_3$)$_2$ **11**
empirical formula	$C_{20}H_{52}Si_6Sn$	$C_{23}H_{55}F_3O_2Si_6Sn$	$C_{30}H_{52}F_{12}O_8Si_6Sn_2$
fw	579.9	707.9	1174.6
cryst size (mm)	0.3 × 0.3 × 0.1	0.4 × 0.3 × 0.3	0.4 × 0.3 × 0.02
cryst system	triclinic	orthorhombic	monoclinic
space group	$P\bar{1}$ (no. 2)	$P2_12_12_1$ (no. 19)	$P2_1/n$ (no. 14)
a (Å)	8.917(1)	9.356(2)	10.046(2)
b (Å	8.966(1)	17.361(3)	9.274(2)
c (Å)	21.502(5)	21.092(3)	26.344(3)
α (deg)	84.29(1)	90	90
β (deg)	89.01(2)	90	92.297(9)
γ (deg)	63.18(1)	90	90
Z	2	4	2
V (Å^3)	1525.8(4)	3426(1)	2452.4(8)
μ (mm^{-1})	1.08	0.99	1.25
θ range (deg)	2−25	2−23	2−25
index range	$0 \le h \le 10$	$0 \le h \le 10$	$0 \le h \le 11$
	$-9 \le k \le 10$	$0 \le k \le 19$	$0 \le k \le 11$
	$-25 \le l \le 25$	$-23 \le l \le 23$	$-31 \le l \le 31$
no. of reflns collected	5372	5422	4560
no. of unique reflns.	5372	4771 (R_{int} 0.031)	4309 (R_{int} 0.086)
no. of reflns with $I > 2\sigma(I)$	4620	4522	2853
$R1$ ($I > 2\sigma(I)$)	0.032	0.034	0.063
$wR2$ (all data)	0.082	0.083	0.172
no. of data/restrts/params	5372/0/244	4771/0/319	4309/0/262
GOF on F^2	1.074	1.046	1.006
abs corr (ψ scan)	T_{max} 1.00, T_{min} 0.90	T_{max} 1.00, T_{min} 0.94	T_{max} 1.00, T_{min} 0.75

Hz) and 0.6 ($^2J_{SiSn} = 45$ Hz, SiMe$_3$), 2.7 ($^2J_{SiSn} = 28$ Hz, SiMe$_2$). ^{119}Sn NMR: δ 148.5 ($^2J_{SnH} = 47$ Hz). IR: 1726 cm^{-1}, ν(CO). MS: m/z 702 (2, M), 693 (15, M − Me), 620 (5, M − SiMe$_4$), 595 (10, M − CO$_2$CF$_3$), 449 (30), 379 (80), 73 (100).

Crystallography. Data were collected on an Enraf Nonius CAD4 diffractometer and corrected for Lorentz and polarization effects and absorption. SHELXS(86 or 97) or SHELXL-(93 or 97) programs were used for structure determinations (Details in Table 2). Non-hydrogen atoms were anisotropic and hydrogen atoms were refined in riding mode with $U_{iso} = 1.2U_{eq}$ or $1.5U_{eq}$ for Me groups. The value of U_{eq} for C27 in **6** is anomalously low, but the attached H atoms are visible on a difference map and refine normally. The assignment as a methyl group is consistent with the spectroscopic evidence.

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Supporting Information Available: Tables showing details of crystal structure determinations, atom coordinates, equivalent isotropic displacement factors, bond lengths and angles, and hydrogen coordinates for compounds **4**, **6**, and **11**. This material is available free of charge via the Internet at http://pubs.acs.org.

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