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Solid State and Solution Behavior of Triorganotin(IV) Derivatives Containing the 2,4,6-Tris(trifluoromethyl)benzene and 1,4-Bis(perfluoroisopropoxy)tetrafluorobenzene Ligands: Crystal and Molecular Structures of $2,4,6\text{-}(CF_3)_3C_6H_2Sn(C_6H_5)_3$ and $1,4\text{-}[(CH_3)_3SnOC(CF_3)_2]_2C_6F_4$

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The syntheses of triorganotin(IV) derivatives of 2,4,6-tris(trifluoromethyl)benzene $R_3SnC_6H_2(CF_3)_3$ [R = CH_3 (**1**), n-C_4H_9 (**2**) and C_6H_5 (**3**)] and 1,4-bis(hexafluoroisopropoxy)tetrafluorobenzene, $[R_3SnOC(CF_3)_2]_2C_6F_4$ [R = CH_3 (**4**) and C_6H_5 (**5**)] are achieved metathetically by reacting the corresponding triorganotin chlorides with $Li[2,4,6\text{-}(CF_3)_3C_6H_2]$ or $[LiOC(CF_3)_2]_2C_6F_4$ in diethyl ether/hexane. The solid state structures of compounds **3** and **4** are investigated by single crystal X-ray analysis. Crystals of **3** belong to the orthorhombic space group $Pna2_1$; fw = 631.3, a = 13.882 (3) Å, b = 21.031 (4) Å, c = 8.324 (2) Å, V = 2430.2 (9) Å^3, Z = 4. Compound **4** crystallizes in a monoclinic system having space group $P2_1/n$; fw = 807.7, a = 7.109 (2) Å, b = 16.767 (3) Å, c = 11.370 (2) Å, β = 101.33 (3)°, V = 1328.8 (5) Å^3, Z = 2. The structures are solved by using the direct method and the final refinement by full matrix least square procedures converged at R = 0.034 and R_w = 0.040 for **3**; and R = 0.077 and R_w = 0.073 for **4** by using 2175 and 1230 refections, respectively, with $F > 3\sigma(F)$. While the solid state structures of **3** and **4** show evidence of Sn−F contacts which are less than the Sn−F van der Waals distance of 3.64 Å, variable temperature ^{19}F NMR studies of both **1** and **3** in $CDCl_3$/toluene-d_8 show no evidence for Sn−F bonding interactions. The ^{119}Sn NMR spectrum for **1** in toluene-d_8 is also indicative of four coordinate tin. Intermolecular Sn−F interaction of the trimethyltin moiety in **4** with a fluorine atom of the CF_3 group of a neighboring molecule results in flattening of the CH_3−Sn−CH_3 angle (average = 115.2°) and sheet polymer formation in the solid state.

Introduction

The role of sterically and/or electronically demanding ligands in achieving kinetic stabilization of compounds with unusual oxidation/coordination numbers has attracted much attention from main group/transition metal chemists.[1,2] These sterically demanding groups may be (a) substituted aromatic systems such as the tris(*tert*-butyl)phenyl (supermesityl), 2,4,6-tris(isopropyl)-phenyl, pentamethylcyclopentadiene (Cp*), (b) highly substituted methyl systems, e.g., bis(trimethylsilyl)methyl and tris-(trimethylsilyl)methyl, or (c) bulky imido ligands such as −Nmesityl(B(mesityl)$_2$), $-N(SiMe_2Ph)_2$ and $-N(SiMe_3)_2$. The latter class has been found to be very effective in stabilizing transition metals with low coordination numbers.[3]

The 2,4,6-tris(trifluoromethyl)benzene ligand (R_f), derived from 1,3,5-tris(trifluoromethyl)benzene (R_fH), was first prepared by McBee and Sanford in 1950.[4] Recently, Chambers *et al.*[5] reported an alternate synthetic route to prepare R_fH in 33% yield by reacting SF_4 with benzene-1,3,5-tris(carboxylic acid) at 150 °C. The abundant availability of R_fH following an improved synthesis,[6] which gave yields of ~90%, provided the opportunity to explore the capability of this ligand to stabilize metals with unusual coordination numbers. Reaction of R_fH with n-butyllithium to give R_fLi followed by reaction with the appropriate organometallic halide is a convenient method for the introduction of the R_f ligand. The molecular structures of **3** and **4** show short (less than van der Waals distances) tin−fluorine contacts with the o-CF_3 groups. Both this steric factor, and electronic factors are important for the stabilization of metals with low oxidation/coordination numbers.[2−14] In order to study the nature of the metal−fluorine contacts (i.e., bonding *versus* non-bonding interactions), the solid state and solution behavior of these organotin(IV) compounds containing trifluoromethy groups bound to carbons β to tin using R_f and 1,4-bis-(hexafluoroisopropoxy)tetrafluorobenzene (R_f*) as ligands are examined. The use of ^{119}Sn NMR as a probe for tin−fluorine interactions has been reported for a Sn(II) derivative.[10] In this work, we use the sensitivity of the ^{119}Sn NMR chemical shift to changes in coordination number to probe the environment around Sn(IV).

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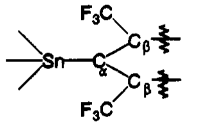
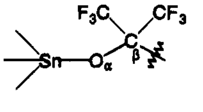
Results and Discussion

The role of *o*-CF_3 groups of the 2,4,6-$(CF_3)_3C_6H_2$ ligand (R_f) in stabilizing a diarylstannylene molecule is demonstrated by the monomeric nature of $Sn(R_f)_2$ in the solid state.[10] Both the X-ray and ^{19}F and ^{119}Sn NMR spectra for the stannylene derivative indicate interactions between the fluorine atoms of the *o*-CF_3 group and the tin nucleus. The X-ray structure of a Sn(IV) compound, $[(R_f)_2SnCl]_2O$, is reported,[8] but there are no low temperature NMR studies. Since ^{119}Sn chemical shifts are very sensitive to changes in coordination number of organotin(IV) compounds, it was expected that this would be a useful tool to probe the local environment at Sn(IV). In addition, we extended this study to the R_f* ligand, where R_f* = $[OC(CF_3)_2]_2C_6F_4$.

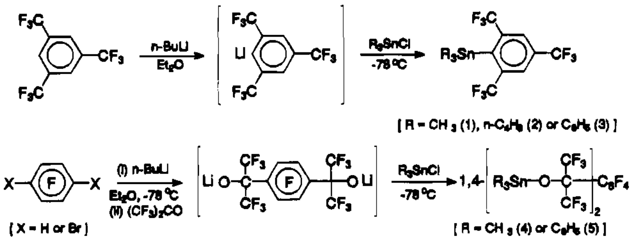


Ligand = Rf



Ligand = Rf*

The reactions of lithiated ligands, R_fLi and R_f*Li, provide easy access to the trifluoromethyl-containing organotin(IV) derivatives, **1**−**5**.



Compounds **1** and **2** are liquids and can be purified by distillation (**1**, bp 54 °C/0.03 Torr) and column chromatography, respectively. Compounds **3**−**5** are solids and they are readily soluble in organic solvents such as diethyl ether, chloroform, dichloromethane, tetrahydrofuran, toluene, etc. Recrystallization of **3** from a diethyl ether/hexane mixture and **4** and **5** from a chloroform/cyclohexane mixture at −20 °C gives these compounds as pure colorless crystalline solids. Single crystal X-ray analyses were obtained on compounds **3** and **4**. Diffraction-quality crystals could not be obtained for **5**.

Crystals for compound **3** belong to a noncentrosymmetric (polar) space group $Pna2_1$, which requires determination of absolute configuration. Due to the presence of the tin atom, which has an effective anomalous scattering effect, the absolute configuration of **3** is easily determined. Refinement of an additional parameter multiplying the imaginary components of the atomic scattering factors (Δf values) indicates that the configuration initially assigned is incorrect. The enantiomorphous configuration is obtained by inversion of the original atomic coordinates. The final value of Roger's parameter[19] η = 0.93 with a small esd value of 12 supports the correctness of the absolute structure reported. This is supported by the lower R and R_w values of 0.0345 and 0.0401 for the absolute structure reported versus values of 0.0364 and 0.0435 for the alternate configuration.

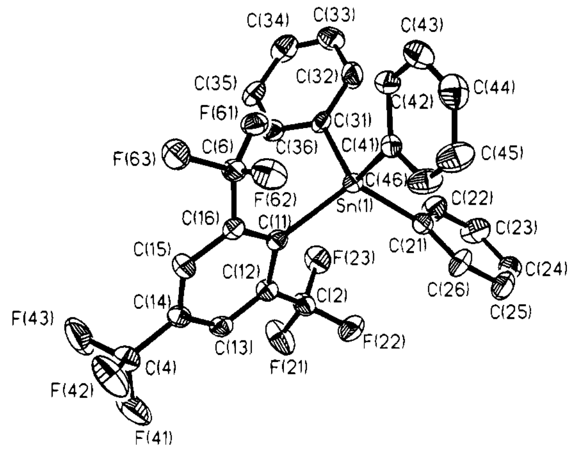


Figure 1. Molecular structure of $(C_6H_5)_3SnR_f$ (**3**).

Table 1. X-ray Crystallographic Parameters for **3** and **4**

empirical formula	$C_{27}H_{17}F_9Sn$	$C_{18}H_{18}F_{16}O_2Sn_2$
fw	631.1	807.7
color, habit	colorless, chunk	colorless, plates
cryst size (mm)	0.15 × 0.40 × 0.50	0.20 × 0.25 × 0.10
cryst syst	orthorhombic	monoclinic
space group	$Pna2_1$	$P2_1/n$
unit cell dimens	a = 13.882(3) Å b = 21.031(4) Å c = 8.324(2) Å	a = 7.109(2) Å b = 16.767(3) Å c = 11.370(2) Å β = 101.33(3)°
volume (Å^3)	2430.2(9)	1328.8(5)
Z	4	2
ϱ_{calc} (g cm^{-3})	1.725	2.019
F(000)	1240	772
abs coeff (mm^{-1})	1.135	2.006
	Data Collection	
2θ range (deg)	3.0−50.0	3.0−55.0
index ranges	$-1 \leq h \leq 16$ $-1 \leq k \leq 24$ $-1 \leq l \leq 09$	$-1 \leq h \leq 09$ $-1 \leq k \leq 21$ $-14 \leq l \leq 14$
no. of data collcd	3210	4163
no. of unique data	2619 (R_{int} = 5.1%)	3044 (R_{int} = 3.94%)
no. of data with $F > 3\sigma(F)$	2175	1229
T_{max}/T_{min}	0.764/0.731	0.789/0.694
	Solution and Refinement	
absolute struct	η = 0.93(12)	
weighting scheme $w^{-1} = \sigma^2(F) + xF^2$	x = 0.0006	x = 0.0010
no. of params refined	335	172
final R indices	R = 3.45% R_w = 4.01%	R = 7.73% R_w = 7.30%
goodness-of-fit	1.00	1.21
largest and mean Δ/σ	0.019, 0.002	0.000, 0.000
data-to-param ratio	6.5:1	7.2:1
largest diff peak (e Å^{-3})	0.57	1.30
largest diff hole (e Å^{-3})	−0.38	−0.92

The structure of **3** is shown in Figure 1. The hydrogen atoms are omitted for clarity. The final atomic coordinates and equivalent isotropic displacement coefficients, and bond distances and bond lengths are listed in Tables 2 and 3, respectively. The monomeric nature of this compound is evident from the stereoscopic packing diagram (Figure 2). Although the geometry around the tin atom is essentially tetrahedral (average C−Sn−C = 109.5°), angles C(11)−Sn(1)−C(21) [115.0° (3)] and C(31)−Sn(1)−C(41) [114.0° (3)] deviate significantly from regular tetrahedral geometry. The Sn(1)−C(11) bond distance of 2.244 (7) Å is greater than the Sn(1)−C(21), Sn(1)−C(31) and Sn(1)−C(41) distances of 2.151 (8), 2.127 (7) and 2.145

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Table 2. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Coefficients ($Å^2 \times 10^3$) for **3**

	x	*y*	*z*	*U*(eq)[a]
Sn(1)	8114(1)	8222(1)	10024	40(1)
C(11)	6765(5)	8674(3)	9078(9)	39(2)
C(12)	6732(5)	9005(3)	7596(10)	41(2)
C(2)	7608(6)	9099(4)	6589(11)	55(3)
F(21)	7500(4)	9571(3)	5541(8)	98(3)
F(22)	7846(4)	8587(3)	5773(7)	78(2)
F(23)	8384(3)	9257(3)	7486(8)	69(2)
C(13)	5888(6)	9255(3)	7024(9)	46(3)
C(14)	5040(5)	9205(4)	7887(10)	44(2)
C(4)	4134(6)	9497(4)	7222(12)	60(3)
F(41)	4161(4)	9634(4)	5708(9)	122(3)
F(42)	3385(4)	9132(3)	7339(12)	108(3)
F(43)	3919(6)	10033(3)	7909(13)	148(4)
C(15)	5044(5)	8910(3)	9350(11)	47(2)
C(16)	5891(4)	8649(3)	9924(14)	42(2)
C(6)	5818(6)	8366(4)	11601(10)	49(3)
F(61)	6595(3)	8465(2)	12491(6)	58(2)
F(62)	5669(4)	7742(3)	11557(8)	76(2)
F(63)	5083(3)	8608(3)	12428(8)	82(2)
C(21)	9155(6)	7964(4)	8224(10)	48(2)
C(22)	10031(6)	8267(5)	8178(14)	72(4)
C(23)	10731(7)	8104(5)	7087(14)	82(4)
C(24)	10580(7)	7615(5)	6044(12)	70(4)
C(25)	9719(8)	7299(4)	6059(13)	73(4)
C(26)	9032(7)	7464(4)	7154(11)	59(3)
C(31)	8741(5)	8921(3)	11547(10)	42(2)
C(32)	9499(6)	8761(5)	12578(13)	70(4)
C(33)	9891(7)	9223(6)	13566(14)	80(4)
C(34)	9578(7)	9829(6)	13512(14)	75(4)
C(35)	8841(6)	10005(4)	12493(12)	62(3)
C(36)	8435(6)	9543(4)	11505(11)	53(3)
C(41)	7792(5)	7342(3)	11212(11)	44(2)
C(42)	7898(7)	7268(4)	12867(11)	59(3)
C(43)	7731(8)	6684(5)	13552(14)	78(4)
C(44)	7440(7)	6175(5)	12684(15)	71(4)
C(45)	7312(8)	6247(5)	11095(15)	81(4)
C(46)	7515(6)	6820(4)	10358(13)	68(4)

[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized $\mathbf{U}_{ij}$ tensor.

(8) Å, respectively, indicating elongation of the Sn−C bond due to the sterically demanding tris(trifluoromethyl)benzene moiety. As a consequence of steric effects, the internal angle around the ipso-carbon in the R_f ring is compressed to 115.3°. The Sn(1)−C(11) distance in **3** [2.244 (7) Å] is between those found in $[(R_f)_2SnCl]_2O$ (average = 2.194 Å) and $Sn(R_f)_2$ (average = 2.281 Å).

Table 3. Selected Bond Lengths (Å) and Bond Angles (deg) for **3**

Bond Lengths

Sn(1)−C(11)	2.244(7)	Sn(1)−C(21)	2.151(8)
Sn(1)−C(31)	2.127(7)	Sn(1)−C(41)	2.145(8)
C(11)−C(12)	1.417(11)	C(12)−C(2)	1.490(11)
C(2)−F(21)	1.330(11)	C(2)−F(22)	1.316(11)
C(2)−F(23)	1.351(10)	C(6)−F(61)	1.325(9)
C(6)−F(62)	1.330(10)	C(6)−F(63)	1.332(10)

Bond Angles

C(11)−Sn(1)−C(21)	115.0(3)	C(11)−Sn(1)−C(31)	104.9(3)
C(21)−Sn(1)−C(31)	108.3(3)	C(11)−Sn(1)−C(41)	110.7(3)
C(21)−Sn(1)−C(41)	104.1(3)	C(31)−Sn(1)−C(41)	114.0(3)
Sn(1)−C(11)−C(12)	122.7(5)	Sn(1)−C(11)−C(16)	122.0(6)
C(12)−C(11)−C(16)	115.3(7)	C(11)−C(12)−C(2)	121.9(6)
C(11)−C(12)−C(13)	121.3(7)	C(2)−C(12)−C(13)	116.8(7)
C(12)−C(2)−F(21)	112.1(7)	C(12)−C(2)−F(22)	112.7(7)
F(21)−C(2)−F(22)	107.5(7)	C(12)−C(2)−F(23)	111.9(7)
F(21)−C(2)−F(23)	105.6(7)	F(22)−C(2)−F(23)	106.6(7)
C(11)−C(16)−C(15)	123.1(9)	C(11)−C(16)−C(6)	122.2(7)
C(15)−C(16)−C(6)	114.6(7)	C(16)−C(6)−F(61)	113.4(6)
C(16)−C(6)−F(62)	111.8(7)	F(61)−C(6)−F(62)	107.2(6)
C(16)−C(6)−F(63)	112.1(6)	F(61)−C(6)−F(63)	106.0(7)
F(62)−C(6)−F(63)	105.8(7)		

The structure of **3** shows two intramolecular tin−fluorine contacts—one from each of the *o*-CF_3 groups. The distances, Sn(1)−F(23) and Sn(1)−F(61) lie at 2.972 (11) and 3.074 (10) Å, respectively, and are lower than the Sn−F van der Waals distance of 3.640 Å.[20] The remaining structural features are not affected by these Sn−F interactions i.e., the angles C(11)−C(16)−C(6) and C(11)−C(12)−C(2) are greater than 120° [122.7 (7) and 121.9 (6)°, respectively]. The C−F bonds involved in forming intramolecular Sn−F contacts vary inversely with their angles, i.e., C(2)−F(23) [1.351 (10) Å] > C(6)−F(61) [1.329 (9) Å], but C(12)−C(2)−F(23) [111.9° (7)] < C(16)−C(6)−F(61) [113.4° (6)]. These contacts may, therefore be regarded as sterically controlled. The *p*-CF_3 group, which has no intra- or intermolecular tin contact, is disordered as shown by larger thermal ellipsoids (Figure 1).

The molecular structure of **4** is shown in Figure 3 as thermal ellipsoids at 30% probability. The final atomic coordinates and equivalent isotropic displacement coefficients are listed in Table 4. The most striking feature of the structure is the highly flattened CH_3−Sn−CH_3 angles, i.e., C(1)−Sn(1)−C(3) = 114.7 (8), C(1)−Sn(1)−C(2) = 113.9 (8) and C(3)−Sn(1)−C(2) = 116.9 (8)°, resulting in appreciable deviation from tetrahedral geometry. Although shortening of the Sn−O bond distance

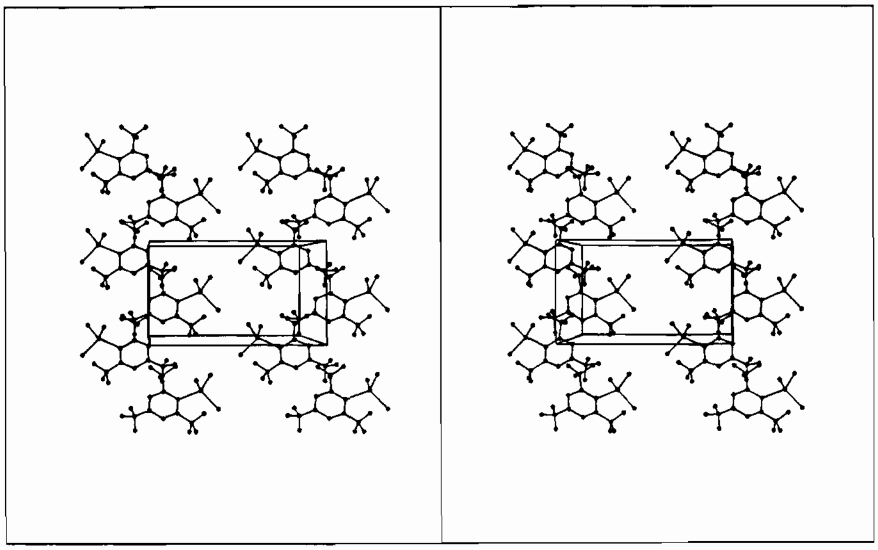


Figure 2. Packing of **3** in a crystal lattice.

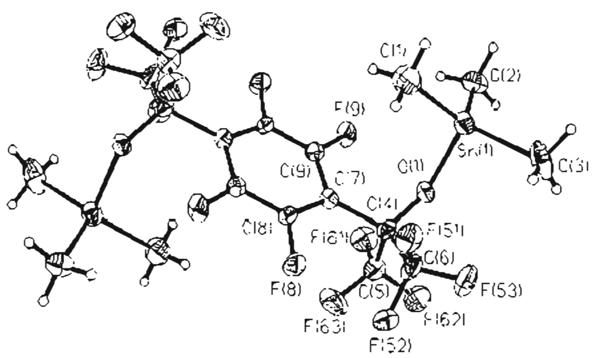


Figure 3. Molecular structure of $[(CH_3)_3SnOC(CF_3)_2]_2C_6F_4$ (**4**).

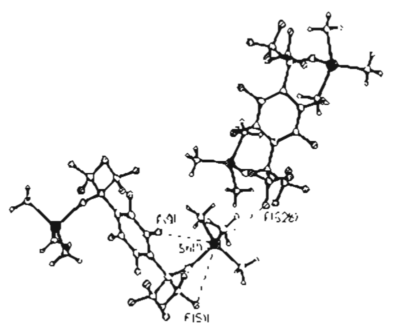
Table 4. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Coefficients ($Å^2 \times 10^3$) for **4**

	x	*y*	*z*	*U*(eq)[a]
Sn(1)	2179(2)	2471(1)	4102(1)	49(1)
O(1)	659(15)	1672(6)	2951(9)	40(4)
C(1)	3152(30)	1895(12)	5756(15)	77(9)
C(2)	−33(27)	3294(11)	4129(16)	66(8)
C(3)	4396(29)	2823(12)	3188(21)	84(9)
C(4)	725(23)	873(10)	2810(13)	38(6)
C(5)	2676(30)	676(12)	2444(16)	57(8)
C(6)	−941(33)	616(12)	1770(17)	62(8)
C(7)	416(20)	403(9)	3948(13)	32(5)
C(8)	1189(22)	−311(9)	4361(12)	36(5)
C(9)	−848(21)	711(9)	4638(13)	36(5)
F(51)	4106(14)	679(6)	3397(9)	64(4)
F(52)	2741(17)	−23(6)	1886(9)	70(5)
F(53)	3078(16)	1219(7)	1678(9)	77(5)
F(61)	−2579(16)	883(7)	1971(9)	72(5)
F(62)	−688(17)	921(7)	741(9)	77(5)
F(63)	−1104(18)	−164(7)	1655(9)	83(5)
F(8)	2460(14)	−697(6)	3805(8)	54(4)
F(9)	−1796(14)	1383(5)	4349(8)	53(4)

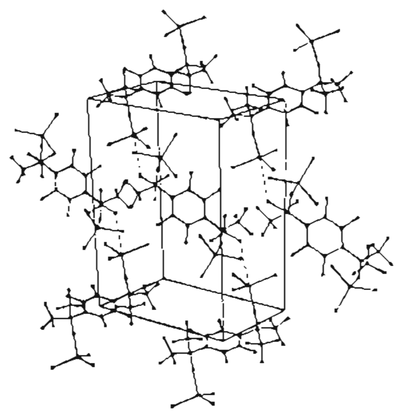
[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Table 5. Selected Bond Lengths (Å) and Bond Angles (deg) for **4**

	Bond Lengths		
Sn(1)−O(1)	2.031(10)	Sn(1)−C(1)	2.107(17)
Sn(1)−C(2)	2.097(19)	Sn(1)−C(3)	2.134(23)
C(4)−O(1)	1.351(19)	C(4)−C(6)	1.561(24)
C(4)−C(5)	1.560(28)	C(4)−C(7)	1.567(22)
C(5)−F(51)	1.332(20)	C(5)−F(52)	1.338(22)
C(5)−F(53)	1.328(23)	C(6)−F(61)	1.309(26)
C(6)−F(62)	1.322(23)	C(6)−F(63)	1.318(24)
	Bond Angles		
C(1)−Sn(1)−C(3)	114.7(8)	C(1)−Sn(1)−C(2)	113.9(8)
C(3)−Sn(1)−C(2)	116.9(8)	C(1)−Sn(1)−O(1)	108.2(6)
C(3)−Sn(1)−O(1)	102.6(6)	C(2)−Sn(1)−O(1)	97.9(6)
O(1)−C(4)−C(6)	109.0(13)	O(1)−C(4)−C(5)	107.1(14)
C(6)−C(4)−C(5)	108.8(14)	O(1)−C(4)−C(7)	112.7(13)
C(6)−C(4)−C(7)	106.2(13)	C(5)−C(4)−C(7)	113.0(13)
Sn(1)−O(1)−C(4)	134.8(9)	C(4)−C(6)−F(61)	110.0(15)
C(4)−C(6)−F(63)	112.8(15)	F(61)−C(6)−F(63)	107.0(18)
C(4)−C(6)−F(62)	110.4(17)	F(61)−C(6)−F(62)	107.6(15)
F(63)−C(6)−F(62)	108.7(16)	C(4)−C(5)−F(52)	115.2(16)
C(4)−C(5)−F(53)	110.3(15)	F(52)−C(5)−F(53)	104.9(15)
C(4)−C(5)−F(51)	111.0(15)	F(52)−C(5)−F(51)	107.2(15)
F(53)−C(5)−F(51)	107.7(16)	C(4)−C(7)−C(8)	127.9(14)
C(4)−C(7)−C(9)	119.1(13)		



Intra- and intermolecular Sn···F contacts



Unit cell packing diagram

Figure 4. A diagram showing the long range Sn···F contacts in **4**.

[2.031 (10) Å] in **4** compared to the Sn−O covalent bond (2.14 Å) indicates some back-donation of electron density from the oxygen atom into the empty d-orbitals of tin, with nonfluorinated alkoxides (where even shorter Sn−O bond lengths are observed) the absence of π-back donation has been suggested.[21] In the case of **4**, the tertiary perfluoroalkoxy ligand may be sufficiently electronegative[22] to prevent back donation of electron density. The Sn(1)−O(1)−C(4) angle is quite large [138.4° (9)] in **4** and the trimethyltin groups are bent away from the two trifluoromethyl groups in order to minimize electronic/steric repulsions. When viewed through the axis passing through the $-(CF_3)_2C-$ carbon along the plane which contains the perfluorobenzene ring, the molecule adopts a staggered configuration to minimize repulsions of four $-CF_3$ and two $(CH_3)_3Sn-$ groups.

There are two Sn−F intramolecular contacts in **4** per tin atom that are less than the van der Waals distance. The two interactions result from a fluorine atom of one of the propylidine $-CF_3$ groups, i.e., Sn(1)−F(51) = 3.462 (11) Å, and from one of the aromatic fluorine atoms, Sn(1)−F(9) = 3.422 (10) Å, as shown in Figure 4. A possible third interaction, Sn(1)−F(53) = 3.62 (11) Å, is at the van der Waals distance within experimental error. All of the intramolecular Sn−F contacts in **3** and **4** are longer than those found in $Sn(R_f)_2$ (2.745 Å).[10] In addition to tin−fluorine interactions, there are several intramolecular F−F contacts which lie in the range from 2.501 to 2.669 Å. These distances, F(8)−F(9), 2.518 (14) Å; F(8)−F(52), 2.501 (14) Å; F(8)−F(51), 2.669 (15) Å are less than the sum of the van der Waals radii of fluorine atoms (~2.68 Å).[23]

The flattening of the trimethyltin moiety can be rationalized by looking at the packing diagram in a unit cell of **4** (Figure

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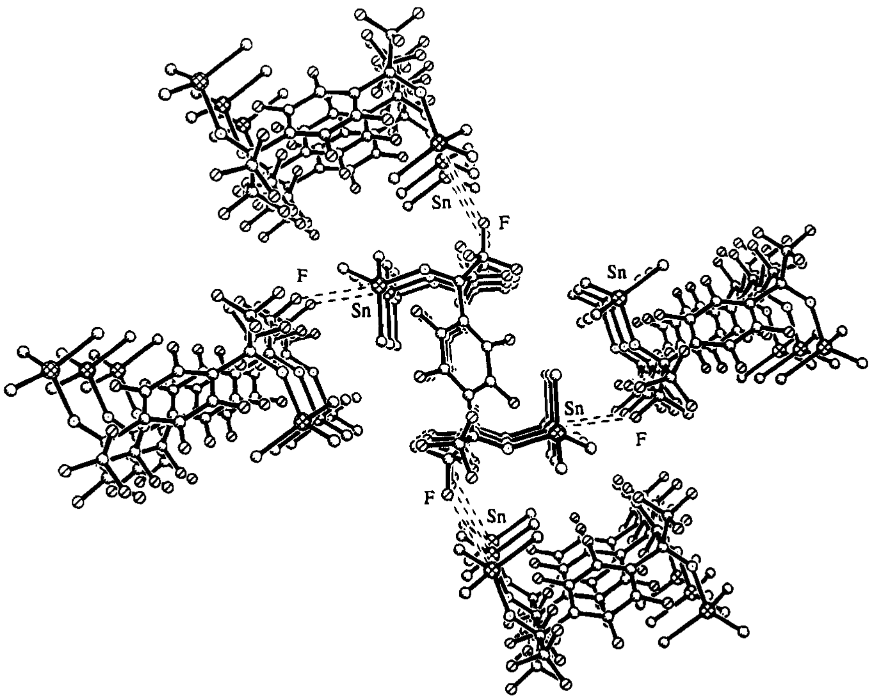

Figure 5. A 3-dimensional perspective view of a crystal lattice of **4** along the *x*-axis. The polymeric sheets lie along the *yz* plane.

4). The CF_3 group not involved with intramolecular Sn–F interactions plays an important role in forming intermolecular Sn–F contacts, i.e. Sn(1)–F(62b) [3.453 Å (11)] (Figure 4). Due to the presence of an inversion center in the molecule, there are two donor sites and two acceptor sites, which connect each of the molecules to four neighboring molecules in the crystal lattice as in Figure 5. This figure shows a three-dimensional perspective view along the x-axis with the polymeric sheets lying along the *yz* axes. The geometry around the tin atom is pseudo trigonal bipyramidal. The linearity of the F(62b)··Sn(1)–O(1) bond [170.3° (3)] is consistent with the fact that electronegative atoms occupy the axial positions in a TBP arrangement.[24]

In order to determine if the tin–fluorine interactions seen in the solid state (i.e., tin–fluorine distances < the sum of the van der Waals radii) take place in solution, ^{1}H, ^{13}C, and variable temperature ^{19}F and ^{119}Sn NMR spectral behavior for **1** were recorded and variable temperature ^{19}F NMR data were obtained for compound **3**. The ^{1}H and ^{13}C NMR of $(CH_3)_3SnR_f$ (**1**) show a septet for both the methyl protons and carbons as well as the tin satellites, *via* coupling to the six *o*-CF_3 fluorine atoms. These peak multiplicities change from a septet to a singlet in the ^{1}H NMR and from a septet to a quartet in the ^{13}C NMR with ^{19}F decoupled. The ^{119}Sn NMR spectrum shows a broad peak with some structural features. The ^{19}F NMR spectra for **1** and **3** show two signals in a 2:1 ratio due to the *o*-CF_3 and *p*-CF_3 fluorine atoms at −58.14 and −63.62 ppm (compound **1**) and, −56.69 and −63.6 ppm (compound **3**), respectively.

In variable temperature ^{19}F NMR experiments, lowering of the sample temperature from 295 to 195 K (at which temperature precipitation occurs) for compounds **1** and **3** does not give rise to splitting of the *o*-CF_3 peak. This indicates the magnetic equivalence of the ortho trifluoromethyl fluorine atoms in solution on the NMR time scale. Similar observations are reported in other cases where metal–fluorine interactions in the solid state are studied.[7,9,13] Such splitting is, however, found in the case of a sterically crowded $Bi(R_f)_3$ derivative.[17] The chemical shifts of both the *o*-CF_3 and *p*-CF_3 resonances for **1** and **3** are found to be temperature dependent. These values decrease to −57.56 and −62.55 ppm, and −56.17 and −62.67 ppm respectively with a decrease in temperature from 295 to 195 K. In the stannylene system,[10] there is a temperature dependent effect on the tin–fluorine coupling constants when toluene is used as a solvent. The ^{19}F NMR spectrum for **1** shows anomolous behavior in the resonance due to the *p*-CF_3 moiety at temperatures between 225 and 213 K. Two resonances appear that are separated by ~6 Hz. This effect is not understood, but may be due to the formation of interactive bonds with solvent molecules. The ^{119}Sn NMR data for **1** do not change between 225 and 213 K. The low temperature ^{119}Sn NMR spectral data for **1** in toluene-d_8 show only a minor shift of $\delta(^{119}Sn)$ from 3.54 ppm to −1.76 ppm. Similarly, in the case of the ^{7}Li NMR spectrum of $[Li\text{-}1,3,5\text{-}(CF_3)_3C_6H_2{\cdot}Et_2O]_2$,[7] no change in multiplicity is observed on lowering the temperature. These observations indicate that in solution, there is no significant perturbation of the nuclear spin of tin by interaction with fluorine atoms. The ^{119}Sn chemical shifts for **1**–**5** lie in a region characteristic of tetracoordinated organotin(IV) compounds.[25,26]

It is also found that the $\delta(^{119}Sn)$ values in $CDCl_3$ for both **4** and **5** i.e., 180.9 and −70.2 lie well below those for compounds **1** (−0.1 ppm) and **3** (−131.4), respectively, which reflects the relative electronegativity of the R_f and R_f^* ligands. In comparison to some other $(CH_3)_3Sn$ derivatives containing halide/pseudohalide ligands, $\delta(^{119}Sn)$ for R_f^* is at lower field than the resonance for CF_3SO_3[27] or Cl/Br/I derivatives,[28] but about 70 ppm upfield from the resonances found for $Me_3SnN(SO_2X)_2$ (X = F,[29] CF_3[27]). Calculation of the average methyl-tin-methyl angles[30] in solution for compound **4** from the $|^{2}J(^{119}Sn-^{1}H)|$ (57.6 Hz, $\theta = 110.8°$) and $|^{1}J(^{119}Sn-^{13}C)|$ (391.5

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Hz, $\theta = 111.1°$) values, obtained from the ^{1}H and ^{13}C NMR spectra, respectively, show a decrease from those found in the solid state (~115.2°). This fact is indicative of a breakdown of the solid state polymeric structure and solvation of monomeric species in solution.

Conclusions

The compounds prepared in this study are characterized by X-ray crystallography (**3** and **4**) and by multinuclear NMR. While tin−fluorine contacts exist in the solid state, the solution behavior shows no such interactions. It appears that the interactions found in the solid state exist predominantly because of steric reasons *vis-a-vis* electronic factors. The 1,4-(hexafluoroisopropoxy)tetrafluorobenzene ligand apparently has the capability to stablize low oxidation/coordination number metals in a manner similar to tris(trifluoromethyl)benzene.

Experimental Section

The reagents, R_3SnCl ($R = CH_3$, C_4H_9 and C_6H_5), *n*-C_4H_9Li (~1.6 M solution in hexane) (Aldrich Chemical Co. Inc.), $CF_3C(O)CF_3$ (Daikin), and 2,4,6-$(CF_3)_3C_6H_3$ and 2,3,5,6-$C_6H_2F_4$ (PCR) were used as received. The compounds Me_3SnR_f (**1**)[5] and 1,4-$(LiOC(CF_3)_2)_2C_6F_4$ (**6**)[18] were prepared according to literature methods. Solvents used are analytical grade and are dried over sodium-benzophenone ketal prior to use. Hexafluoroacetone is quantitated in a conventional vacuum line equipped with a Heise Bourdon tube and Televac thermocouple gauges using *PVT* techniques. Infrared spectra are recorded on a Perkin−Elmer 1710 FT-IR spectrophotometer as neat solids/liquids/Nujol mulls between KBr disks. Room temperature ^{1}H, ^{13}C, ^{19}F and ^{119}Sn NMR spectra are obtained as $CDCl_3$ solutions on a Bruker AC300 FT-NMR spectrometer operating at 300.31 (^{1}H), 75.47 (^{13}C), 282.41 (^{19}F) and 111.92 (^{119}Sn) MHz, respectively. The ^{119}Sn NMR spectra are recorded by inserting a ~1 mm sealed capillary containing neat $(n\text{-}C_4H_9)_3SnCl$ that serves as an external reference (144.0 ppm) while the ^{1}H, ^{13}C and ^{19}F chemical shifts are referenced to $(CH_3)_4Si$ (0 ppm), $CDCl_3$ (77.0 ppm) and $CFCl_3$ (0 ppm), respectively. The $CH_3-Sn-CH_3$ (θ, average) angles in solution state are calculated from published mathematical relationships.[30] The variable temperature ^{19}F and ^{119}Sn NMR spectra are recorded over a temperature range of 295 to 195 K using $CDCl_3$/toluene-d_8 as solvent. Mass spectra are obtained on a Varian VG 7070 HS mass spectrometer and the peaks reported correspond to the ^{120}Sn isotope. Fragments are identified by theoretical simulation of the isotopic pattern. Elemental analyses are performed by Beller Microanalytisches Laboratorium, Göttingen, Germany. Melting points (uncorrected) are obtained by using a Mel-temp apparatus. Manipulations and measurements involving *n*-butyllithium are performed in a dry nitrogen atmosphere.

Preparation of R_3SnR_f [$R = CH_3$ (1),[5] C_4H_9 (2) and C_6H_5 (3); $R_f = C_6H_2(CF_3)_3$)]. In a typical preparation, a stirred solution of R_fH (~5 mmol) in diethyl ether is lithiated by the dropwise addition of an equimolar amount of *n*-butyllithium at 25 °C. After the addition is complete, the reaction mixture is stirred for an additional hour. The reaction mixture is then cooled to −78 °C and a solution of R_3SnCl in 10 mL of diethyl ether is transferred into the reaction vessel using a double tipped cannula. The suspension is allowed to warm slowly to 25 °C, stirred overnight and refluxed for 2 h before the addition of water. The organic layer is separated, washed twice with water and finally dried over $MgSO_4$. Removal of volatile materials and subsequent distillation for **1**, chromatography for **2** or cooling the concentrated solution for **3** at −20 °C give the desired R_3SnR_f as pure products.

Properties of 2,4,6-$(CF_3)_3C_6H_2Sn(CH_3)_3$ (1). This compound is isolated as a colorless liquid in 69% yield. The spectral data obtained and not previously reported are as follows. IR (cm^{-1}) (neat liquid): 3100 w, 2992 mw, 2923 ms, 1625 s, 1573 mw, 1440 w br sh, 1386 ms, 1284 vs, 1263 vs, 1191 vs, 1137 vs, 1086 s, 915 vs, 854 vs, 838 ms, 785 s, 737 ms, 723 m, 691 vs, 671 ms, 561 w, 539 ms. NMR (ppm): ^{1}H[5], δ 0.46 [sept, 9H, $(CH_3)_3Sn$, $^{2}J(^{119}Sn-^{1}H) = 56$ Hz, $\theta = 110°$], 8.1 [2H, $C_6H_2(CF_3)_3$]; ^{13}C, δ −3.8 [sept, $(CH_3)_3Sn$, $^{1}J(^{119}Sn-^{13}C) = 375.8$ Hz, $\theta = 109.7°$, $^{5}J(^{19}F-^{13}C) = 3.6$ Hz], 123.8 [q, p-CF_3, $^{1}J(^{19}F-^{13}C) = 272.2$ Hz], 124.0 [q, *o*-CF_3, $^{1}J(^{19}F-^{13}C) = 274.2$ Hz], 125.6 (br s, C_m), 131.6 [q, C_p, $^{2}J(^{19}F-^{13}C) = 34.2$ Hz}, 139.4 [q, C_o, $^{2}J(^{19}F-^{13}C) = 30.9$ Hz, $^{2}J(^{119}Sn-^{13}C_o) = 6.4$ Hz}, 147.5 (s, C_i); ^{13}C {^{19}F}, δ −3.8 [q, $(CH_3)_3Sn$, $J(^{1}H-^{13}C) = 375.8$ Hz}, 123.8 (s, p-CF_3), 124.0 (s, *o*-CF_3), 125.6 (br s, C_m), 131.6 [q, C_p, $^{2}J(^{19}F-^{13}C) = 34.2$ Hz}, 139.4 [q, C_o, $^{2}J(^{19}F-^{13}C) = 30.9$ Hz, $^{2}J(^{119}Sn-^{13}C_o) = 6.4$ Hz], 147.5 (s, C_i); ^{19}F, δ −58.2 (s, 6F, *o*-CF_3), −63.8 (s, 3F, p-CF_3) (lit. −58.7 and −64.7, respectively);[5] ^{119}Sn, δ −0.1 ($CDCl_3$), 3.54 (toluene-d_8). MS/EI [*m/e* (species) relative intensity]: 431 ($M^+ - CH_3$) 39.9; 401 ($SnC_6H_2(CF_3)_3^+$) 6.3; 243 ($C_9H_2F_7^+$) 100; 165 ($(CH_3)_3Sn^+$) 8.4; 139 (SnF^+) 10.2.

Properties of 2,4,6-$(CF_3)_3C_6H_2Sn(C_4H_9)_3$ (2). This compound is isolated as a pale yellow liquid in 57% yield. The spectral data obtained are as follows. IR (cm^{-1}) (neat liquid): 2960 vs, 2931 vs, 2874 vs, 1625 ms, 1588 m, 1466 vs, 1381 s, 1282 vs, 1192 vs, 1142 vs, 1085 s, 1040 m, 1002 m, 952 ms, 914 vs, 882 m, 854 ms, 837 m, 737 ms, 688 vs, 670 s, 597 mw, 562 w, 542 w, 512 mw, 452 w, 436 mw. NMR (ppm): ^{1}H, δ 0.89 [t, 3H, $(CH_3C_3H_6)_3Sn$], 1.0−1.6 [m, 6H, $(CH_3C_3H_6)_3$-Sn], 8.1 [2H, $C_6H_2(CF_3)_3$]; ^{19}F, δ −58.5 (s, 6F, *o*-CF_3), −63.7 (s, 3F, p-CF_3); ^{119}Sn, δ −17.2. MS/EI [*m/e* (species) relative intensity]: 515 ($M^+ - C_4H_9$) 50.4; 401 ($SnC_6H_2(CF_3)_3^+$) 9.8; 339 ($C_4H_9C_6H_2(CF_3)_3^+ + 1$) 6.9; 291 ($(C_4H_9)_3Sn^+$) 14.3; 243 ($C_9H_2F_7^+$) 55.3; 177 ($C_4H_9Sn^+$) 19.7; 139 ($SnF^+$) 1.8; 121 ($SnH^+$) 7.4; 57 ($C_4H_9^+$) 100.

Properties of 2,4,6-$(CF_3)_3C_6H_2Sn(C_6H_5)_3$ (3). This compound is found as colorless crystals in 72% yield (crop 1 and 2 combined) (mp 155 °C). The spectral data obtained for **3** are as follows. IR (cm^{-1}): 3071 mw, 3051 mw, 2925 vs, 2855 vs, 1624 m, 1573 w, 1482 m, 1431 ms, 1337 w, 1294 vs, 1283 vs, 1263 ms, 1186 vs, 1142 vs, 1085 m, 1076 ms, 1024 w, 998 mw, 916 m, 853 m, 838 w, 734 s, 693 s, 685 s, 671 w, 618 vw, 564 vw, 513 vw, 460 m, 447 ms, 436 mw, 411 w. NMR (ppm): ^{1}H, δ 7.2−7.5 [m, 15H, $(C_6H_5)_3Sn$], 8.2 [2H, C_6H_2-$(CF_3)_3$]; ^{19}F, δ −56.7 (s, 6F, *o*-CF_3), −63.6 (s, 3F, p-CF_3); ^{119}Sn, δ −131.4. MS/CI [*m/e* (species) relative intensity]: 555 ($M^+ - C_6H_5$) 100; 357 ($C_6H_4C_6H_2(CF_3)_3^+$) 12.1; 351 ($(C_6H_5)_3Sn^+$) 4.4; 243 ($C_9H_2F_7^+$) 27.7; 197 ($C_6H_5Sn^+$) 5.6; 139 (SnF^+) 3.9; 121 (SnH^+) 3.1; 77 ($C_6H_5^+$) 12.7. Anal. Calcd for $C_{27}H_{17}F_9Sn$ (631.1): C, 51.38; H, 2.72; F, 27.1. Found: C, 51.29; H, 2.78; F, 27.4.

Preparation of $[R_3SnOC(CF_3)_2]_2C_6F_4$ (*R* = CH_3 (4) and C_6H_5 (5)]. Under a dry nitrogen atmosphere, a solution of $(CH_3)_3SnCl$ in 10 mL of diethyl ether is added *via* a cannula to a solution of **6** (~5 mmol) in 30 mL of the same solvent at −78 °C with vigorous stirring. The suspension is allowed to warm slowly to 25 °C, stirred overnight and refluxed for two hours before addition of water. The organic layer is separated, washed twice with water and dried over $MgSO_4$. After removal of volatile materials a wax like solid remains. Recrystallization of this material from cyclohexane affords **4** as a colorless crystalline product. The triphenyltin(IV) derivative, **5**, is prepared similarly.

Properties of $[(CH_3)_3SnOC(CF_3)_2]_2C_6F_4$ (4). Compound **4** is isolated as colorless crystals in 37% yield (mp 149 °C). The spectral data obtained are as follows. IR (cm^{-1}): 3009 vw, 2924 w, 2858 w, 1464 vs, 1424 mw, 1319 sh, 1278 ms, 1254 s, 1233 sh, 1225 vs, 1215 vs, 1199 s, 1166 vs, 1146 s, 1017 ms, 987 mw, 940 s, 788 ms, 777 ms, 732 s, 724 s, 697 m, 622 w, 573 w, 550 ms, 524 w, 435 w. NMR (ppm): ^{1}H, δ 0.48 [s, $(CH_3)_3Sn$, $^{2}J(^{119}Sn-^{1}H) = 57.6$ Hz, $\theta = 110.8°$]; ^{13}C, δ −1.8 [s, $(CH_3)_3Sn$, $J(^{119}Sn-^{13}C) = 391.5$ Hz, $\theta = 111.1°$], 116 [C_i], 123.0 [q, CF_3, $^{1}J(^{19}F-^{13}C) = 292$ Hz], 146 [dd, C_mF, $^{1}J(^{19}F-^{13}C) = 268.7$ Hz, $^{2}J(^{19}F-^{13}C) = 15.1$ Hz]; ^{19}F, δ −75.3 (t, 12F, CF_3), −134.3 (br m, 4F, C_6F_4), $^{5}J(^{19}F-^{19}F) = 14.7$ Hz; ^{119}Sn, δ 180.9. MS/EI [*m/e* (species) relative intensity]: 795 ($M^+ - CH_3$) 7.8; 165 ($(CH_3)_3$-Sn^+) 100; 139 (SnF^+) 12.3; 121 (SnH^+) 1.4. Anal. Calcd for $C_{18}H_{18}F_{16}O_2Sn_2$ (807.4): C, 26.75; H, 2.23; F, 37.7. Found: C, 26.98; H, 2.28; F, 37.0.

Properties of $[(C_6H_5)_3SnOC(CF_3)_2]_2C_6F_4$ (5). Compound **5** is found as colorless crystals in 23% yield (mp 188−189 °C). The spectral data obtained are as follows. IR (cm^{-1}): 3072 w, 3055 m, 2908 vs, br, 1581 w, 1463 vs, 1433 vs, 1337 m, 1279 vs, 1256 vs, 1224 vs, 1167 vs, 1149 vs, 1077 s, 1021 s, 998 s, 983 s, 781 m, 729 vs, 698 vs, 664 m, 620 vw, 551 ms, 542 w, 528 vw, 449 s, 403 w. NMR (ppm): ^{1}H, δ 7.2−7.8 [m, $(C_6H_5)_3Sn$]; ^{19}F, δ −78.7 (t, 12F, CF_3), −136.8 (br m, 4F, C_6F_4), $^{5}J(^{19}F-^{19}F) = 14.9$ Hz; ^{119}Sn, δ −70.2. MS/CI [*m/e* (species) relative intensity]: 1086 ($M^+ - C_6H_5 - F$) 23.1; 713 ($M^+ - (C_6H_5)_3Sn - C_2F_5 + 1$) 100. Anal. Calcd for $C_{48}H_{30}F_{16}O_2Sn_2$

(1179.4): C, 48.84; H, 2.54; F, 25.8. Found: C, 48.99; H, 2.84; F, 25.4.

Crystal Structure Solution and Refinement. The crystallographic data for **3** and **4** are collected on a Siemens R3m/V diffractometer (upgraded to P4) at 298 K using graphite monochromated Mo K_α radiation ($\lambda = 0.71073$ Å). The lattice parameters are refined from 40 for **3** and 34 for **4** well centered reflections with $10 < 2\theta < 30$. The diffraction data are collected using 2θ-ω scans, with online profile fitting and variable scan speeds. Three standard reflections (146, 225 and 233, for **3**; $15\bar{1}$, 060 and 031, for **4** are monitored during data collection. There is about a 15% decrease in these reflections for **4** over the data collection period indicating some decomposition. Data are corrected for Lorentz/polarization effects and for absorption (empirical ψ scans). The structures are solved by the direct method using Shelx86[31] and SHELXTL Plus and refined by full matrix least squares analysis to minimize the quantity $\sum w(F_o - F_c)^2$. All non-H atoms are refined anisotropically and hydrogen atoms are included in calculated positions using a riding model with a fixed isotropic U value. The absolute determination of **3** is carried out making use of the anomalous dispersion of the tin atom. The values for η, R and R_w obtained after the last cycle of refinement are 0.93, 0.0345, and 0.0401, respectively. The correct crystallographic configuration is obtained by inversion of initial coordinates.

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Supplementary Material Available: For compounds **3** and **4**, tables listing full data collection and processing parameters, bond lengths and bond angles, atomic coordinates, equivalent isotropic and anisotropic displacement coefficients, and hydrogen atom coordinates and isotropic displacement coefficients (16 pages). Ordering information is given on any current masthead page.

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