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Stepwise fluorination of $[MeAlN(2,6\text{-}i\text{-}Pr_2C_6H_3)]_3$ using trimethyltin fluoride as fluorinating agent

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Dedicated to Professor Alan H. Cowley for his outstanding contributions in Chemistry

Abstract

The fluorination of $[MeAlN(2,6\text{-}i\text{-}Pr_2C_6H_3)]_3$ **1** using 1 and 2 eq., respectively, trimethyltin fluoride leads to the mono- and difluoro compounds, $[FAl(MeAl)_2(N(2,6\text{-}i\text{-}Pr_2C_6H_3))_3]\cdot 2THF$ **2** and $[(FAl)_2MeAl(N(2,6\text{-}i\text{-}Pr_2C_6H_3))_3]\cdot 3THF$ **3**, where each methyl group can be selectively exchanged for terminal fluorine atoms (Al–F). The reaction of **1** and 3 eq. of trimethyltin fluoride leads to the trifluoro compound $[FAlN(2,6\text{-}i\text{-}Pr_2C_6H_3)]_3\cdot 3THF$ **4** and $[Me_2SnN(2,6\text{-}i\text{-}Pr_2C_6H_3)]_2$ **5** as a by-product. The core of compound **5** consists of a tin–nitrogen four-membered Sn_2N_2 ring.

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Keywords: Aluminum; Stepwise fluorination; Tin

1. Introduction

Since 1955, Ziegler and Köster [1,2] had pioneered the preparation of dialkylaluminum fluorides, only a small number of other alkylaluminum fluorides [3–7] has been synthesized due to the lack of suitable fluorination routes. General attempts of selective fluorination have led to product mixtures.

Recently, Uhl et al. reported that the reaction of a carbaalane cluster $(AlEt)_7(C{\equiv}CHC_6H_5)_2(CCH_2C_6H_5)_3H$ with $HBF_4\cdot OEt_2$ yields the fluorine derivative $(AlEt)_7(C{\equiv}CHC_6H_5)_2(CCH_2C_6H_5)_3(\mu_3\text{-}F)$ [8]. In the past we have reported the facile preparation of groups 4–6 and main group fluorides [9–11] from their corresponding chlorides using trimethyltin fluoride as fluorinating reagent. Furthermore, based on this experience we have developed in recent years the chemistry of aminoalane mono- and difluorides [12] and trisylaluminum difluorides (trisyl $=$ $(Me_3Si)_3C$–) [13] using trimethyltin fluoride in methyl-fluorine exchange reactions with the concomitant generation of volatile tetramethyltin. However, a stepwise fluorination has not been realized until now.

Herein, we report the first controlled stepwise fluorination of $[MeAlN(2,6\text{-}i\text{-}Pr_2C_6H_3)]_3$ **1** [14] with trimethyltin fluoride in THF solution. However, the fluorination of **1** with 3 eq. of trimethyltin fluoride yields a mixture of $[FAlN(2,6\text{-}i\text{-}Pr_2C_6H_3)]_3\cdot 3THF$ **4** and a four-membered tin–nitrogen Sn_2N_2 ring **5** in a molar ratio of 10:1.

2. Results and discussion

The reactions of trimethyltin fluoride in various stoichiometries with **1** in THF solution at room temperature leads to the colorless iminoalane fluoride-THF adducts $[FAl(MeAl)_2(N(2,6\text{-}i\text{-}Pr_2C_6H_3))_3]\cdot 2THF$ **2** and $[(FAl)_2MeAl(N(2,6\text{-}i\text{-}Pr_2C_6H_3))_3]\cdot 3THF$ **3**, respectively, having terminal Al–F bonds (Scheme 1). Increasing the amount to 3 eq. of trimethyltin fluoride at room temperature leads to the trifluoro iminoalane **4** and to a tin–nitrogen four-membered ring **5** (Scheme 2). In contrast to our previously reported aminoalane difluorides [12], which could be prepared in either toluene or THF solution, the reactions of **1** with trimethyltin fluoride in toluene or *n*-hexane gave only mixtures of several compounds as shown by NMR spectroscopy. Compounds **2**–**4** can be isolated in an analytically pure form after recrystallization from *n*-hexane (**2**, **3**) and

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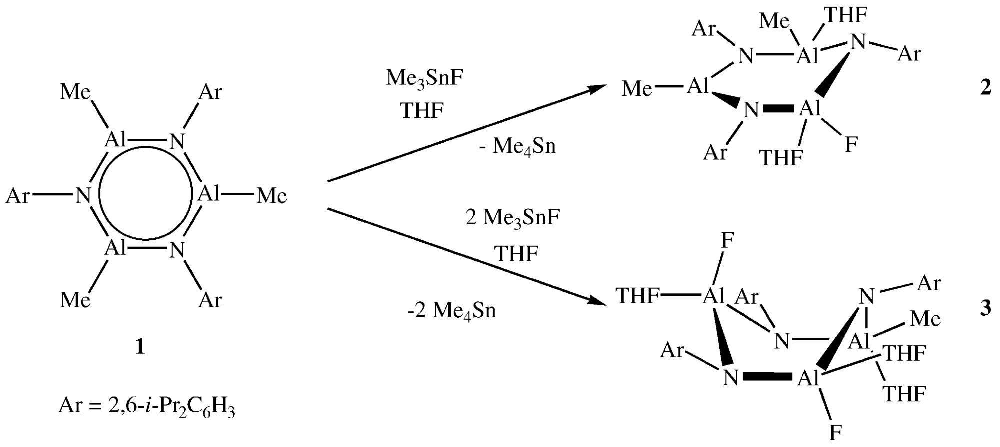
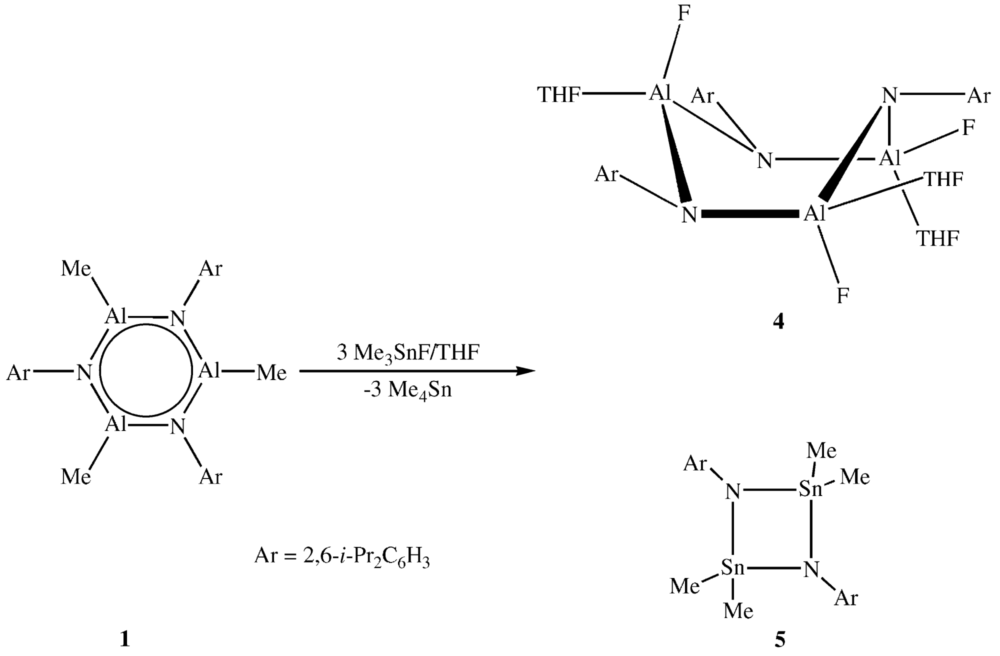


Scheme 1.



Scheme 2.

THF/*n*-hexane (**4**), respectively, in high yields (95% **2** and **3**, 70% **4**). Crystals of compound **5** were obtained from THF/toluene (1:10) by cooling the solution (0 °C). Compounds **2**–**5** were characterized by elemental analysis and mass spectrometry, and NMR spectroscopic measurements. The molecular structures of **2**, **4** and **5** have been determined by X-ray crystallography.

The structures of compounds **2** and **4** contain a six-membered Al_3N_3 ring system. The crystal structure of **2** is shown in Fig. 1 and the bond lengths and angles are given in Table 1. The six-membered ring of compound **2** adopts an envelope conformation with N(2) pointing up with respect to the plane which is formed by the remaining five atoms of the ring (mean deviation from the best plane 0.02 Å), while N(2) is 0.49 Å out of this plane. Upon coordination of THF molecules to Al(1) and Al(2) the two atoms loose their planarity, and their coordination spheres are distorted tetrahedrally with bond angles ranging from 104.1 to 116.4°. Thus, the Al–N-bonds of **2** become slightly longer compared to those of the starting material **1** [14]. Surprisingly, Al(3) is not coordinated by a THF molecule. Therefore, the bonds between Al(3) and N(1) or N(3) are slightly shorter compared with the other Al–N-distances in **2**. The environment of Al(3) is planar within the experimental error (mean deviation from the plane 0.01 Å) with bond angles between 119.9 and 120.9°. The Al–F distance in **2** is 1.67 Å which is comparable to those found in the literature [13].

The structure of **4** is shown in Fig. 2 and selected bond lengths and angles are given in Table 2. The Al_3N_3 six-membered ring of compound **4** has a boat conformation with

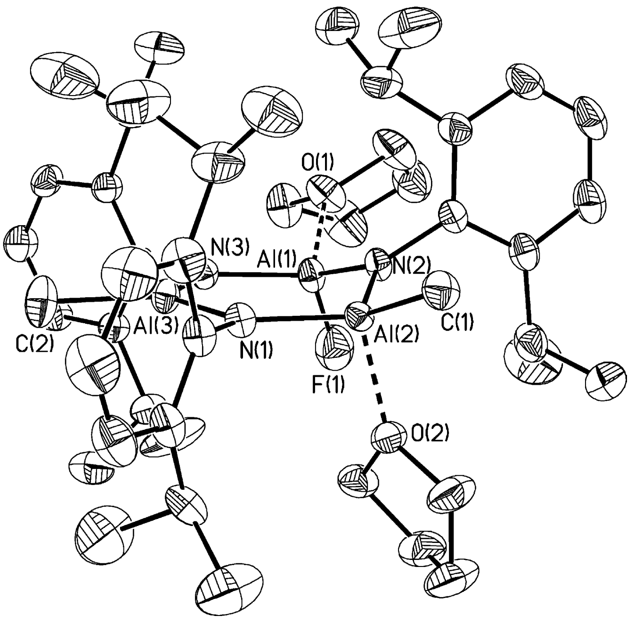


Fig. 1. Molecular structure of **2**.

Table 1
Selected bond lengths (Å) and angles (°) for **2**

Al(1)–F(1)	1.672(2)	Al(1)–N(2)–Al(2)	123.7(1)
Al(1)–N(2)	1.813(2)	Al(1)–N(3)–Al(3)	121.5(1)
Al(1)–N(3)	1.818(2)	Al(2)–N(1)–Al(3)	122.1(1)
Al(2)–N(1)	1.840(2)	N(1)–Al(3)–N(3)	119.91(9)
Al(2)–N(2)	1.834(2)	N(1)–Al(2)–N(2)	110.78(8)
Al(3)–N(1)	1.789(2)	N(2)–Al(1)–N(3)	111.45(8)
Al(3)–N(3)	1.795(2)		

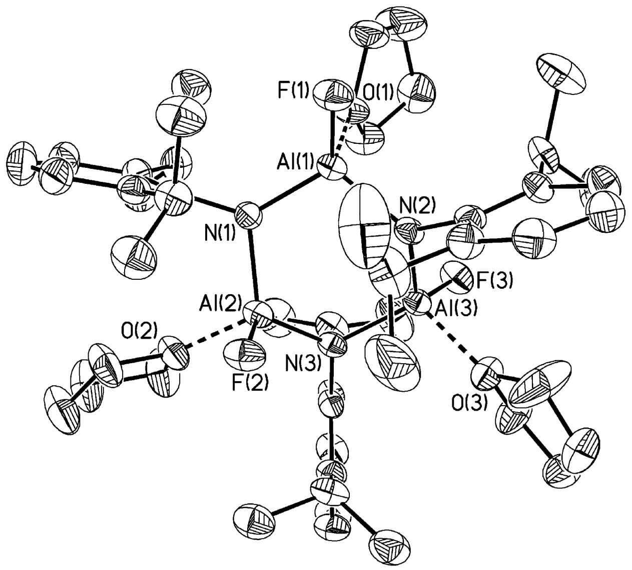


Fig. 2. Molecular structure of **4**.

the stern pointing up at Al(2). Due to the coordination of THF molecules to the Al atoms, the ring looses its planarity and all Al–N bonds become slightly longer than those in **1**. Two of the terminal fluorine atoms are located above and one below with respect to the ring arrangement (Fig. 2). In the boat conformation, all i-$Pr_2C_6H_3$ groups can adopt the favorable equatorial position. In **4**, the terminal Al–F bonds range from 1.680 to 1.688 Å and are somewhat longer than those in [{(2,6-i-$Pr_2C_6H_3$)N($SiMe_3$)AlF_2}$_3$] (1.634–1.642 Å) [12] and AlF_3 (1.63 Å) [15], as determined by electron diffraction but comparable with those in [$(Me_3Si)_3CAlF_2$]$_3$ (1.657–1.681 Å) [13]. The distorted tetrahedral coordination sphere of each Al atom is completed by one THF molecule.

Table 2
Selected bond lengths (Å) and angles (°) for **4**

Al(1)–F(1)	1.6798(17)	F(1)–Al(1)–N(1)	119.64(10)
Al(2)–F(2)	1.6821(17)	F(1)–Al(1)–N(2)	114.07(10)
Al(3)–F(3)	1.6879(16)	F(2)–Al(2)–N(1)	111.97(9)
Al(1)–N(1)	1.808(2)	F(2)–Al(2)–N(3)	117.46(10)
Al(1)–N(2)	1.833(2)	F(3)–Al(3)–N(2)	114.79(9)
Al(2)–N(1)	1.831(2)	F(3)–Al(3)–N(3)	114.33(9)
Al(2)–N(3)	1.818(2)	Al(1)–N(1)–Al(2)	113.84(12)
Al(3)–N(2)	1.813(2)	Al(1)–N(2)–Al(3)	119.94(11)
Al(3)–N(3)	1.815(2)	Al(2)–N(3)–Al(3)	114.51(11)
		N(1)–Al(1)–N(2)	110.88(10)
		N(1)–Al(2)–N(3)	114.69(10)
		N(2)–Al(3)–N(3)	110.39(10)

The ^{19}F NMR spectrum of **4** in benzene-d_6 (25 °C) shows two singlets (δ −166.3 and 153.9 ppm); they integrate in a 2:1 ratio, corresponding to two different kinds of terminal fluorine atoms. In the ^{19}F NMR spectra of **2** and **3** only one resonance (**2**: δ −154.9; **3**: δ −158.7 ppm) is observed for the fluorine atoms.

The molecular structure of **5** is shown in Fig. 3. Selected bond lengths and angles are given in Table 3. Compound **5** exists as a dimer. The EI–MS spectrum of **5** shows as well the molecular fragment [M^+] (m/z 648), as [$M^+/2$] (m/z 324). The core of compound **5** is a four-membered Sn_2N_2 ring. The Sn coordination spheres are distorted tetrahedral with bond angles ranging from 81.75 to 116.2°. Interestingly, the N–Sn–N angle (81.75°) is smaller than the Sn–N–Sn angle (98.25°). The Sn–N distances (2.047 and 2.059 Å) are comparable to those found in [t-Bu_2Sn-N-t-Bu]$_2$ (2.059 and 2.055 Å) and [t-Bu_2SnNSO_2Me]$_2$ (2.085 and 2.092 Å) [16].

A possible mechanism for the formation of **5** is that one molecule of compound **4** reacts with an additional molecule of Me_3SnF forming difluoro aluminum anion and the

Table 3
Selected bond lengths (Å) and angles (°) for **5**

Sn(1)–N(1)	2.059(2)	N(1)–Sn(1)–N(1A)	81.75(9)
Sn(1)–N(1A)	2.047(2)	N(1)–Sn(1)–C(13)	112.93(11)
Sn(1)–C(13)	2.139(3)	N(1a)–Sn(1)–C(13)	116.27(11)
Sn(1)–C(14)	2.137(3)	N(1)–Sn(1)–C(14)	114.15(10)
		N(1A)–Sn(1)–C(14)	114.34(11)
		C(14)–Sn(1)–C(13)	113.66(12)
		Sn(1)–N(1)–Sn(1A)	98.25(9)

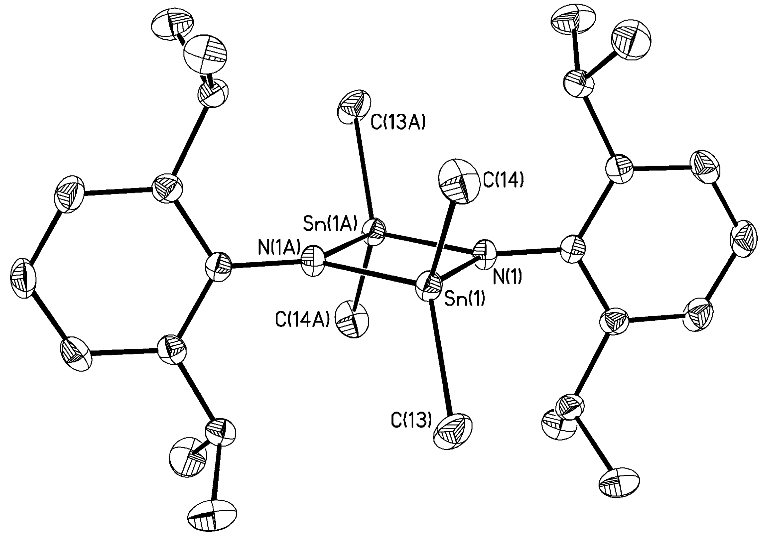


Fig. 3. Crystal structure of **5**.

Me_3Sn^+ cation. A stable Me_3Sn^+ cation can be found in solution by photoassisted electron transfer from R_4Sn to an acceptor [17]. The tin atom forms a bond to the nitrogen and after elimination of a Me group the monomer of **5** is formed. As in $[(F_3C)_3C_6H_2]_2SnNMes$ [18] the stannanimine dimerizes to give **5**.

In conclusion, we have accomplished the synthesis of three novel iminoalane fluorides **2**–**4**. For the first time we observed a stepwise fluorination of **1**. In addition the fluorination of **1** with 3 eq. of Me_3SnF leads to a four-membered Sn_2N_2 ring system. Obviously a high concentration of fluorine atoms in an Al–N molecule leads to the partial decomposition of the molecule.

3. Experimental

All experiments were performed using standard Schlenk techniques under a dry nitrogen atmosphere, due to the sensitivity of the reactants and products towards air and moisture. All solvents were distilled from sodium/benzophenone and degassed prior to use. $[MeAlN(2,6\text{-}i\text{-}Pr_2C_6H_3)]_3$ **1** was prepared as described in the literature [14]. Elemental analyses were performed by the Analytisches Labor des Instituts für Anorganische Chemie der Universität Göttingen. NMR spectra were recorded on Bruker AM 200, Bruker Avance 200, Bruker AM 250, Bruker AS 400 and Bruker Avance 500 spectrometers and were externally referenced to Me_4Si, Me_4Sn and $CFCl_3$. Melting points were measured in sealed glass tubes.

*3.1. Synthesis and characterization of $[FAl(MeAl)_2(N(2,6\text{-}i\text{-}Pr_2C_6H_3))_3]\cdot 2THF$, **2***

A solution of **1** (980 mg, 1.5 mmol) in THF (30 ml) was added to a suspension of Me_3SnF (272 mg, 1.5 mmol) in THF (40 ml). The mixture was stirred for 12 h at room temperature, until the entire solid had dissolved. The solvent was removed in vacuo and the residue was treated with *n*-hexane (25 ml), affording **2** as a colorless solid. Yield: 1.14 g (95%). mp: 230 °C. ^1H NMR (200 MHz, C_6D_6): −0.84 (s, 6H, $AlCH_3$), 0.80 (m, 4H, THF), 1.05 (m, 4H, THF), 1.41 (m, 36H, $CH(CH_3)_2$), 3.25 (m, 4H, THF), 4.30 (m, 6H, $CH(CH_3)_2$), 7.05 (m, 9H, aromat. H). ^{19}F NMR (235 MHz, C_6D_6): −154.9 (s). EI–MS *m/z* (rel. int.): 728 [M^+–THF] (5), 162 [ArH] (100). Elemental analyses for $C_{46}H_{73}Al_3FN_3O_2$: anal. calcd. for C, 69.06; H, 9.12; F, 2.37; found C, 68.3; H, 9.0; F, 2.6%.

*3.2. Synthesis and characterization of $[(FAl)_2MeAl(N(2,6\text{-}i\text{-}Pr_2C_6H_3))_3]\cdot 3THF$, **3***

A solution of **1** (980 mg, 1.5 mmol) in THF (30 ml) was added to a suspension of Me_3SnF (544 mg, 3.0 mmol) in THF (40 ml). The mixture was stirred for 12 h at room temperature, until the solid was dissolved completely. The solvent was removed in vacuo and the residue was treated with *n*-hexane (25 ml), affording **3** as a colorless solid. Yield: 1.25 g (95%). mp: 231 °C. ^1H NMR (400 MHz, C_6D_6): −0.74 (s, 3H, $AlCH_3$), 0.83 (m, 8H, THF), 1.24 (m, 4H, THF), 1.41 (m, 36H, $CH(CH_3)_2$), 3.25 (m, 8H, THF), 3.80 (m, 4H, THF), 4.40 (m, 6H, $CH(CH_3)_2$), 7.15 (m, 9H, aromat. H). ^{19}F NMR (235 MHz, C_6D_6): −158.7 (s). EI–MS *m/z* (rel. int.): 497 [M^+–ArH–3THF] (10), 162 [ArH] (100). Elemental analyses for $C_{49}H_{78}Al_3F_2N_3O_3$: anal. calcd. for C, 67.17; H, 8.97; F, 4.33; found C, 66.6; H, 9.0; F, 4.4%.

*3.3. Synthesis of $[FAlN(2,6\text{-}i\text{-}Pr_2C_6H_3)]_3\cdot 3THF$, **4** and $[Me_2SnN(2,6\text{-}i\text{-}Pr_2C_6H_3)]_2$, **5***

A solution of **1** (1.30 g, 2.00 mmol) in THF (40 ml) was added to a suspension of Me_3SnF (1.11 g, 6.0 mmol)

Table 4
Crystallographic data for **2** and **4**

	Compound	
	2	**4**
Empirical formula	$C_{46}H_{73}Al_3FN_3O_2$	$C_{48}H_{75}Al_3F_3N_3O_3$
Formula weight	800.01	880.05
Temperature (K)	133(2)	200(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	*C*2/*c*	$P2_1/n$
Unit cell dimensions	$a = 31.847(6)$ Å, $b = 18.813(4)$ Å, $c = 18.840(4)$ Å, $\beta = 123.26(3)^\circ$	$a = 12.258(4)$ Å, $b = 21.069(7)$ Å, $c = 19.035(9)$ Å, $\beta = 93.45(3)^\circ$
Volume (Å^3), *Z*	9439(3), 8	491(1), 4
Density (calcd.) (Mg/m^3)	1.126	1.191
Absorption coefficient (mm^{-1})	0.122	0.130
F(0 0 0)	3472	1896
Crystal size	0.5 mm × 0.3 mm × 0.2 mm	1.0 mm × 1.0 mm × 0.8 mm
Θ range for data collection (°)	2.12–27.48	3.53–25.05
Index ranges	$-41 \leq h \leq 34$, $0 \leq k \leq 24$, $0 \leq l \leq 24$	$-14 \leq h \leq 14$, $-1 \leq k \leq 25$, $-22 \leq l \leq 22$
Reflections collected	106,493	9295
Independent reflections	10,809 ($R_{int} = 0.0433$)	8666 ($R_{int} = 0.0511$)
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	10,809/165/530	8666/0/553
Goodness-of-fit on F^2	1.067	1.040
Final *R* indices [$I > 2\sigma(I)$]	$R1 = 0.0602$, $wR2 = 0.1374$	$R1 = 0.0536$, $wR2 = 0.1204$
R indices (all data)	$R1 = 0.0778$, $wR2 = 0.1492$	$R1 = 0.0765$, $wR2 = 0.1393$
Largest diff. peak and hole (eÅ^{-3})	0.976 and −0.748	0.386 and −0.288

in THF (40 ml). The mixture was stirred for 12 h at room temperature, until the solid was dissolved completely. The solvent was removed in vacuo and the residue was treated with *n*-hexane (80 ml), affording **4** and **5** as a mixture of colorless products. The mixture was dissolved in THF/*n*-hexane (30 ml) (1:5) and stored at −26 °C for 2 days to afford colorless crystals of **4**. Crystallization at 0 °C from THF/toluene (1:10) (30 ml) resulted in **5** as a colorless crystalline solid.

3.4. Characterization of compound, 4

Yield: 1.23 g (70%). mp: >300 °C. ^{1}H NMR (250 MHz, C_6D_6): 0.77 (m, 4H, THF), 1.02 (m, 8H, THF), 1.25 (d, 12H, $CH(CH_3)_2$), 1.41 (d, 6H, $CH(CH_3)_2$), 1.50 (d, 6H, $CH(CH_3)_2$), 1.65 (d, 6H, $CH(CH_3)_2$), 1.73 (d, 6H, $CH(CH_3)_2$), 3.20 (m, 4H, THF), 3.55 (m, 4H, THF), 3.83 (m, 4H, THF), 4.40 (m, 4H, $CH(CH_3)_2$), 4.75 (sept, 2H, $CH(CH_3)_2$), 7.00 (m, 9H, aromat. H). ^{19}F NMR (235 MHz, C_6D_6): −166.3 (s, 2F), −153.9 (s, 1F). EI–MS *m*/*z* (rel. int.): 501 [M^+–3THF–ArH] (20), 162 [ArH] (100). Elemental analyses for $C_{48}H_{75}Al_3F_3N_3O_3$: anal. calcd. for C, 65.50; H, 8.59; F, 6.48; found C, 64.8; H, 8.4; F, 6.5%.

3.5. Characterization of compound, 5

Yield: 0.23 g (2.3%). mp: 280 °C. ^{1}H NMR (200 MHz, C_6D_6): 0.40 (s, 12H, $Sn(CH_3)_2$, 1.28 (d, $J = 6.9$ MHz, 24H, $CH(CH_3)_2$), 4.27 (sept, $J = 6.9$ MHz, 4H, $CH(CH_3)_2$), 7.12 (m, 6H, aromat. H). ^{119}Sn NMR (186 MHz, C_6D_6): 114.67 (s). EI–MS *m*/*z* (rel. int.): 648 [M^+] (40), 633 [M^+–Me] (80), 324 [$M^+/2$] (90). Elemental analyses for $C_{28}H_{46}N_2Sn_2$: anal. calcd. for C, 36.63; H, 7.15; N, 4.32; found C, 35.9; H, 7.4; N, 4.5%.

3.6. Crystallography

Single crystals of **2**, **4** and **5** were mounted on a glass fiber under nitrogen gas in a rapidly cooled perfluoropolyether [19]. Diffraction data for **2** was collected on a STOE-Siemens–Huber four-circle diffractometer, **4** on a STOE AED2 four-circle diffractometer and **5** on a STOE IPDS. The structures were solved by direct methods (SHELXS-96) [20] and refined against F^2 using SHELXL-97 [21]. All non-hydrogen atoms were refined anisotropically with similarity and rigid bond restraints on the disordered solvent molecules in the structure of **2**. All hydrogen atoms were included in the refinement in geometrically ideal positions. Crystallographic data for **2**, **4** and **5** are given in Tables 4 and 5.

Crystallographic data (including structure factors for the structures in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC 117616 (**2**), CCDC 185851 (**4**) and CCDC 185850 (**5**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, (fax: +44-1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).

Table 5
Crystallographic data for **5**

Empirical formula	$C_{28}H_{46}N_2Sn_2$
Formula weight	648.08
Temperature (K)	133(2)
Wavelength (Å)	0.71073
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	$a = 9.849(2)$ Å, $b = 8.9882(18)$ Å, $c = 16.879(3)$ Å, $\beta = 100.35(3)^\circ$
Volume (Å^3), Z	1469.8(5), 2
Density (calcd.) (Mg/m^3)	1.464
Absorption coefficient (mm^{-1})	1.716
$F(0\,0\,0)$	656
Θ range for data collection (°)	3.18–24.67
Index ranges	$-11 \leq h \leq 11$, $-10 \leq k \leq 10$, $-19 \leq l \leq 19$
Reflections collected	8205
Independent reflections	2352 ($R_{\text{int}} = 0.0888$)
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2352/0/151
Goodness-of-fit on F^2	1.130
Final R indices [$I>2\sigma(I)$]	$R1 = 0.0252$, $wR2 = 0.0747$
R indices (all data)	$R1 = 0.0262$, $wR2 = 0.0756$
Largest diff. peak and hole (eÅ^{-3})	0.478 and −0.565

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