In search for a pentacoordinated monoorgano stannyl cation

Mikhail S. Nechaev [a,b,*], Oleg V. Chernov [a,b], Ivan A. Portnyagin [a,b], Victor N. Khrustalev [c], Rinat R. Aysin [c], Valery V. Lunin [a,b]

[a] Department of Chemistry, M.V. Lomonosov Moscow State University, Leninskie Gory 1/3, Moscow 119991, Russian Federation
[b] A.V. Topchiev Institute of Petrochemical Synthesis, RAS, Leninsky Prosp., 29, Moscow 119991, Russian Federation
[c] A.N. Nesmeyanov Institute of Organoelement Compounds, RAS, Vavilov Str., 28, Moscow 119991, Russian Federation

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ABSTRACT

Alkylation of $Sn(OCH_2CH_2NMe_2)_2$ (**1**) by MeI or MeOTf leads to a mixture of quaternary ammonium salts by alkylation of the NMe_2 moiety. Reaction of $Sn(acac)_2$ (**2**) with MeOTf gives unexpected redistribution product $Sn(acac)OTf$ (**3**), which is a rare example of mono acetylacetonato tin (II) derivatives. Pentacoordinated monoorgano stannyl cation $PhSn(OCH_2CH_2NMe_2)_2^+$ was generated by salt metathesis from $PhSn(OCH_2CH_2NMe_2)_2Cl$ (**5**) and $Ag[Al(OCH(CF_3)_2)_4]$ or $Ag[B(C_6F_5)_4]$. This cation was not isolated due to its strong electrophilic nature. It abstracts substituents from aluminate and borate weakly coordinating anions (WCAs) leading to redistribution products $[Al[OCH(CF_3)_2]_2OCH_2CH_2NMe_2]_2$ (**6**) and $[Ph(C_6F_5)Sn(OCH_2CH_2NMe_2)_2][H_2OB(C_6F_5)_3]$ (**9**), respectively. Structures of **3** and **6** were established by single-crystal X-ray diffraction analysis.

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1. Introduction

Tricoordinate stannylium (**A**), tetra- and pentacoordinated stannyl cations (**B–F**) are a prominent topic of recent research in the field of organotin chemistry [1,2]. Since these species are heavy analogs of carbocations R_3C^+, there is fundamental interest associated with their stability, bonding patterns and reactivity. In addition, they have received increasing interest in applied research as catalysts for the polymerization of alkenes [3,4], acylation of alcohols [5,6], and the Diels–Alder cycloaddition [7,8].

R - organic substituent
D - donor group
X - inorganic substituent
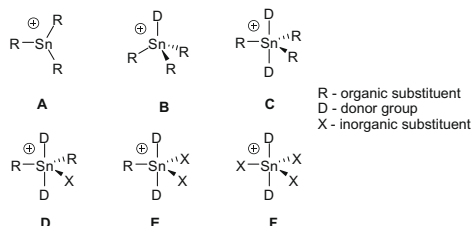
Several stannylium ions **A** were isolated in the solid state [9–13]. In all cases these were triorgano derivatives stabilized by a combination of bulky substituents and the weakly coordinating anion (WCA [14]) $[B(C_6F_5)_4]^-$.

Tri-, di-, and inorgano stannyl cations stabilized by one or two donor groups were isolated in the solid state. The most common type are pentacoordinate triorgano derivatives **C** [15–24]. The use of NCN monoanionic tridentate ligands enables stabilization of stannyl cations even in presence of nucleophilic anions, such as halogenides and pseudohalogenides [18,19,23]. There are only single examples of the types: tetracoordinated triorgano cation **B** [25], pentacoordinate diorgano cation **D** [26], and even fully inorganic pentacoordinate cation **F** [24]. Pentacoordinated monoorgano stannyl cations **E** are still elusive, although several attempts were made at their isolation [3,4,24]. The first hexacoordinated monoorganotin cation $[[PhSn(H_2O)_3-(\mu-OH)]_2][[1,5-C_{10}H_6-(SO_3)_2]_2]$ was recently isolated in solid state [27].

We have recently isolated pentacoordinated monoorgano germyl cations $RGe(OCH_2CH_2NMe_2)_2^+$ (R = Me, Ph) stabilized by two intramolecular N → Ge coordination bonds in the presence of chloride and iodide anions [28,29]. Related tin derivatives $RSn(OCH_2CH_2NMe_2)_2X$ bearing halogenides X (R = aryl, alkyl; X = Cl, I) are covalent compounds [30,31]. In this contribution, we present our recent attempts to isolate the first examples of pentacoordinated monoorgano stannyl cations in the presence of weakly coordinating anions (WCAs).

* Corresponding author. Address: Department of Chemistry, M.V. Lomonosov Moscow State University, Leninskie Gory 1/3, Moscow 119991, Russian Federation. Tel./fax: +7 495 9392677.
E-mail address: nechaev@nmr.chem.msu.ru (M.S. Nechaev).

2. Results and discussion

2.1. Alkylation of Sn(II) compounds

Tin (II) compounds are known to insert into C–X bonds [2]. Thus, if reacted with alkylating agent bearing weakly coordinating anion (WCA) leaving group one might expect an ionic product. Analogously to the synthesis of the monoorgano germyl cation $MeGe(OCH_2CH_2NMe_2)_2^+$ [29] we treated stannylene $Sn(OCH_2CH_2NMe_2)_2$ (**1**) with alkylating agents MeI and MeOTf in dichloroethane (Scheme 1). Mixtures of unidentified products were obtained. We suppose that amino groups are alkylated under these conditions. Several signals corresponding to $–CH_2–NMe_3^+$ fragments of ammonium salts were found in ^1H (at 3.20–3.60 ppm) and ^{13}C (at 55–68 ppm) NMR spectra.

Amino group is a soft Lewis base, which preferentially reacts with soft Lewis acids such as alkyl halogenides and carbocations (Pearson's HSAB principle [32]). Thus, the exchange of a soft amino group to a harder oxygen containing group might shift the reactivity towards alkylating agents from harder ligand to softer tin atom. Indeed, it was previously found that $Sn(acac)_2$ (**2**, acac = acetylacetonate) reacts with organic bromides and iodides to give $RSn(acac)_2X$ (X = Br, I; R = alkyl, benzyl, etc.) [33–36]. The intermediate formation of ion pairs $[RSn(acac)_2]^+X^-$ was observed [33].

We attempted to obtain ionic compound $[RSn(acac)_2]^+X^-$ by replacement of the halide by weaker coordinating triflate. Interaction of $Sn(acac)_2$ (**2**) with MeOTf in ethylene dichloride gave divalent tin compound $Sn(acac)OTf$ (**3**, Scheme 2). Therefore, as for the reaction of stannylene **1** with MeX (X = I or OTf), methylation of the ligand, but not the tin atom, takes place. $Sn(acac)OTf$ (**3**) was isolated as colorless crystals soluble in chloroform, benzene and dichloromethane. The structure of **3** was unambiguously established by single-crystal X-ray diffraction analysis (Fig. 1).

It is important to note that complex **3** is the fourth β-diketonate derivative of tin(II) and only the second mono-β-diketonate-substituted compound (beyond $Sn(ac)(acac)$ (**4**) [37], the structure of which has been determined by X-ray diffraction study. Unlike the structure of **4** which contains isolated $Sn(ac)(acac)$ molecules, complex **3** is a coordination polymer, with the tin atoms bridged by bidentate triflate ligands (Fig. 1, Table S1). The triflate ligands are asymmetrically disposed relative to the tin atoms, with the intermolecular coordination Sn–O distances of 2.395(3) and 2.587(3) Å. The tin atoms in **3** can be regarded as being at the centre of a distorted trigonal bipyramid, with the lone electron pair occupying an equatorial site.
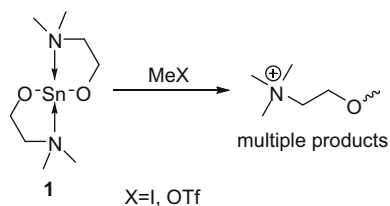
Scheme 1.
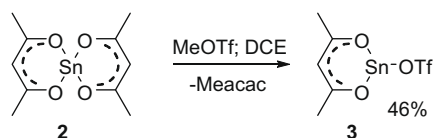
Scheme 2.

2.2. Salt metathesis with Ag[WCA]

Since our attempts to obtain stannyl cations via alkylation of tin (II) compounds failed we tackled an alternative approach, i.e. replacement of the chloride in $PhSn(OCH_2CH_2NMe_2)_2Cl$ (**5**) by a weakly coordinating anion (WCA). Metathesis of halogenides with alkali metal or silver salts of WCAs is a convenient way of stabilization of electrophilic and coordinatively unsaturated cations [14]. The most common and easily accessible WCAs are fluoroalkoxy aluminates $[Al(OR^F)_4]^-$ (R^F = $CH(CF_3)_2$, $C(CF_3)_3$) [38,39], and fluoroaryl borates $[BAr_4^F]^-$ (Ar^F = C_6F_5 [40,41], 3,5-$(CF_3)_2C_6H_3$ [42–45]).

Upon addition of $Ag[Al(OCH(CF_3)_2)_4]$ to a solution of $PhSn(OCH_2CH_2NMe_2)_2Cl$ (**5**) in dichloromethane immediate precipitation of AgCl takes place. After filtration and concentration of the reaction mixture white crystals of **6** were obtained (Scheme 3). The structure of **6** was established by single-crystal X-ray diffraction analysis (Fig. 2).

Apparently, the formation of **6** proceeds via intermediate formation of stannyl cation in **7** (Scheme 3). Electrophilic monoorgano stannyl cation in **7** abstracts a OR^F group from the aluminate anion yielding **8** and $Al(OR^F)_3$. Further alkoxy groups redistribution gives the final dimeric aluminium complex **6**. Formation of intramolecular N → Al coordination bonds in **6** facilitates this process. The tin containing products were not isolated. ^1H NMR spectrum of the mother liquors contains signals of several Ph and $OCH_2CH_2NMe_2$ groups. We suppose that the formation of a mixture of redistribution tin products of general formula $Sn(OCH_2CH_2NMe_2)_x(OR^F)_yPh_z$ ($x + y + z = 4$) is occurring [30,31].

Compound **6** is dimeric in the solid state, bridged by the oxygen atoms of the dimethylaminoethoxy ligand (Fig. 2, Table S2). The coordination number of aluminum is five and the coordination geometry corresponds to a distorted trigonal bipyramid with the coordinating oxygen and nitrogen atoms in the axial positions. The equatorial positions are occupied by two oxygen atoms of the 1,3-hexafluoro-2-propanolato ligands and the bridging alkoxide oxygen atom of the dimethylaminoethoxy ligand. The shortest Al–O distances are found for the aluminum-propanolato bonds (1.721(5)–1.740(5) Å) which are very close to the values of analogous bonds in the related compounds [46–54]. The central Al_2O_2 ring is perfectly planar. The two Al–O distances within the ring are different (Al–O_{eq} 1.841(5) and Al–O_{ax} 1.896(5) Å). This is a typical feature of five-coordinate dimeric aluminum alkoxides [55–61]. The Al–N distances (2.080(6)–2.093(7) Å) and the O_{ax}–Al–N bond angles (157.5(2)–158.2(2)°) are similar to the values found for $[(4-BrC_6H_4O)_2AlOCH_2CH_2N(CH_3)_2]_2$ (2.097(9) Å/157.0(4)°) [58].

The alternative approach explored for the isolation of stannyl cation $PhSn(OCH_2CH_2NMe_2)_2^+$ was the use of perfluoro borate $[B(C_6F_5)_4]^-$. Addition of $Ag[B(C_6F_5)_4]$ to a dichloromethane solutions of $PhSn(OCH_2CH_2NMe_2)_2Cl$ have led to the precipitation of AgCl. After filtration and concentration to dryness of the reaction mixture, an off-white powder of **9** (Scheme 4) was obtained in good yield ~90%. Unfortunately, we were unable to obtain X-ray quality crystals since the product tended to form oily mixtures upon dissolution and foams upon concentration in different solvent mixtures. Nevertheless, the product was characterized by ^1H, ^{13}C, ^{11}B, ^{19}F and ^{119}Sn NMR.

All spectra were clean which is evident for single reaction product. Only one set of signals corresponding to $OCH_2CH_2NMe_2$ and Ph groups were found in ^1H and ^{13}C spectra. Two methyl groups in NMe_2 are equivalent at room temperature, which is not characteristic for ionic compounds [28,29]. Thus, it was proposed that the product have covalent nature. Indeed, the ^{119}Sn signal is virtually unchanged (−395.6 ppm) in comparison to initial chloride $PhSn(OCH_2CH_2NMe_2)_2Cl$ (−395.2 ppm), although it should appear at a lower field if a cation was formed [1]. In ^{13}C and ^{19}F spectra there are two sets of C_6F_5 groups. One set of relative intensity 3
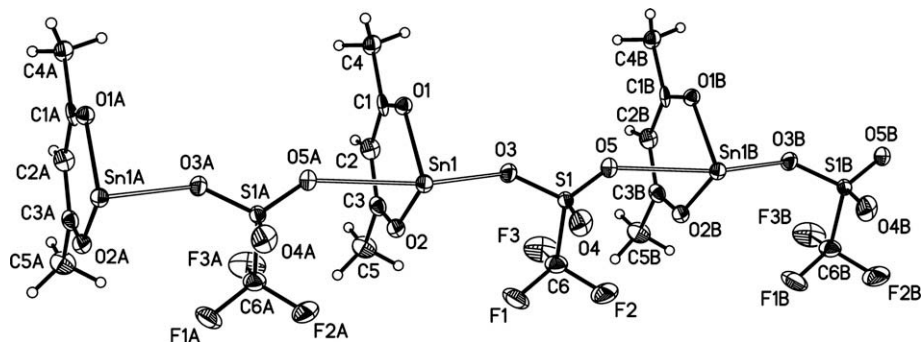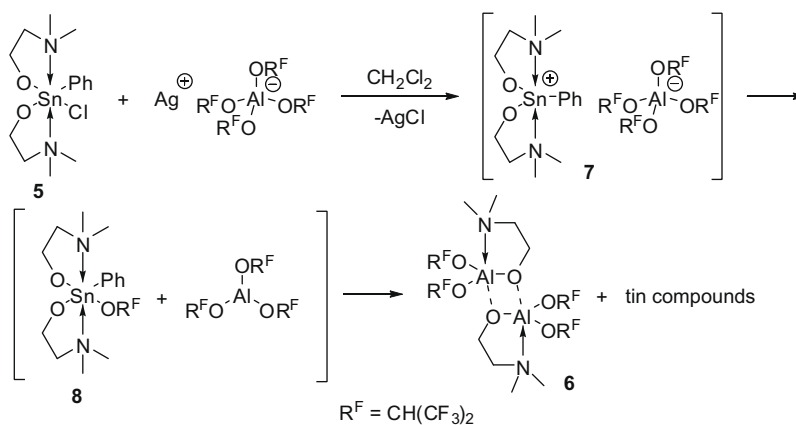
Fig. 1. Crystal structure of coordination polymer **3** (ellipsoids are drawn at 50% probability level). Double lines indicate coordination bonds.

$R^F = CH(CF_3)_2$

Scheme 3.

corresponds to $B(C_6F_5)_3$ moiety. The other set of intensity 1 corresponds to the C_6F_5 group attached to tin. This is also supported by J_{SnF} and J_{SnC} coupling in $Sn-C_6F_5$ moiety. ^{11}B signal at -16.85 ppm is characteristic for tetracoordinated boron atom.

The IR spectrum of **9** in Nujol exhibits a very broad band in the region 3500–2000 cm^{-1}, which can be assigned as ν_{OH} stretching of OH groups involved in a strong hydrogen bond. It corresponds to

interaction of protic acid $H_2O-B(C_6F_5)_3$ with amino groups of $Ph(C_6F_5)Sn(OCH_2CH_2NMe_2)_2$. Most likely, $H_2O-B(C_6F_5)_3$ was formed from $B(C_6F_5)_3$, initially formed in the reaction, which abstracted water from imperfectly dried solvent during workup.

Thus, as for aluminate $[Al(OR^F)_4]^-$, strongly electrophilic penta-coordinated monoorgano stannyl cation $PhSn(OCH_2CH_2NMe_2)_2^+$ in **10** abstracts a substituent from borate $[B(C_6F_5)]^-$ to give covalent
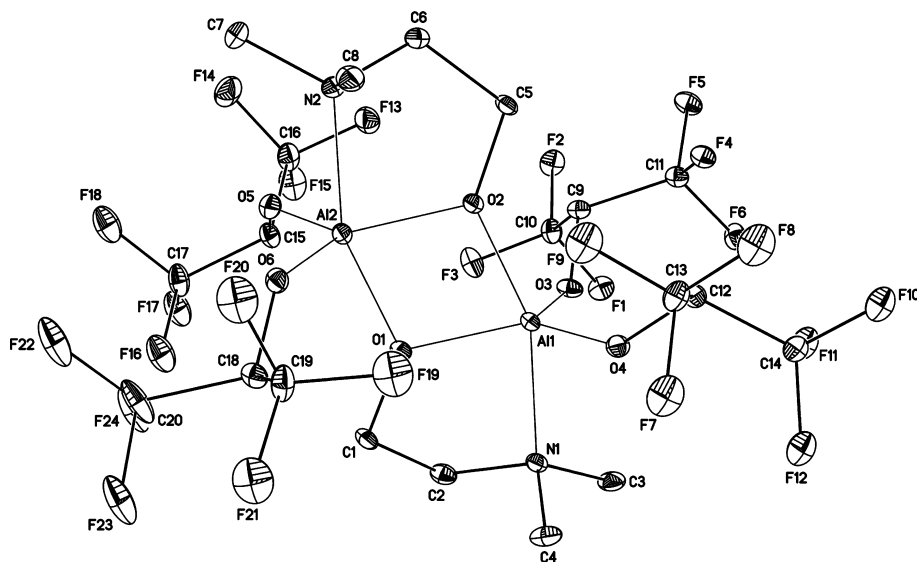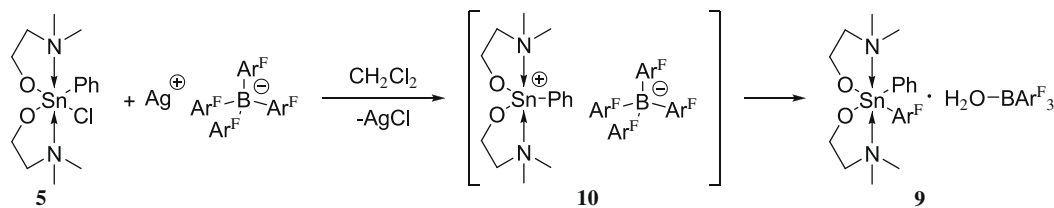

Fig. 2. Molecular structure of dimer **6** (ellipsoids are drawn at 30% probability level). One of the two crystallographically independent molecules is shown. The disordered fluorine and all hydrogen atoms are omitted for clarity.

Scheme 4.

tin compound [Ph(C_6F_5)Sn(OCH$_2$CH$_2$NMe$_2$)$_2$][H$_2$OB(C_6F_5)$_3$] **9** (Scheme 4). Ligand abstraction by strong electrophiles is one of the previously documented limitations of WCAs [14].

3. Conclusions

In our attempts to isolate the first pentacoordinated monoorgano stannyl cation we have found that stannylene Sn(OCH$_2$CH$_2$NMe$_2$)$_2$ (**1**) is alkylated at the amino groups. Contrary to the previously reported alkylation of the tin atom in Sn(acac)$_2$ (**2**) by alkyl halides giving tin (IV) compounds, **2** reacted with MeOTf to give unexpected heteroleptic stannylene Sn(acac)OTf (**3**), which is a rare example of mono acetylacetonato tin (II) derivative. The monoorgano stannyl cation PhSn(OCH$_2$CH$_2$NMe$_2$)$_2^+$ generated by salt metathesis from PhSn(OCH$_2$CH$_2$NMe$_2$)$_2$Cl and Ag[WCA] is a very strong electrophile. It was not isolated due to abstraction of anions (OR^F or C_6F_5) from fluorinated aluminate and borate WCAs. Thus, future attempts at isolating pentacoordinated monoorgano stannyl cations should take this pitfall into account. The design of stabilized RSn$^{(IV)}$X$_2^+$ cations (e.g. by increasing bulk around the tin atom) and/or more robust WCAs might provide a solution to this problem.

4. Experimental

4.1. General procedures

All manipulations were carried out under a purified argon atmosphere using standard Schlenk techniques. The commercially available solvents were purified by conventional methods and distilled immediately prior to use [62]. PhSn(OCH$_2$CH$_2$NMe$_2$)$_2$Cl [30], AgAl(OCH(CF$_3$)$_2$)$_4$ [38,39], AgB(C_6F_5)$_4$ [41] and Sn(AcAc)$_2$ [63] were synthesized as described earlier.

NMR spectra were recorded on an "AVANCE-400" NMR at 400.13 MHz (^{1}H) and 100.62 MHz (^{13}C). Chemical shifts are indirectly referenced to TMS via the solvent signals. ^{11}B (128.38 MHz), ^{19}F (376.50 MHz) and ^{119}Sn (149.21 MHz) NMR spectra were referenced externally to BF$_3$·Et$_2$O, CFCl$_3$ and SnMe$_4$, respectively. Melting points were measured in an sealed capillary using a SANYO Gallenkamp PLC melting point apparatus without any additional corrections. Elemental analyses were performed on a Carlo Erba EA1108 CHNS-O elemental analyzer. IR spectrum was recorded using a Carl Zeiss Specord M82 spectrophotometer with resolution of 2 cm^{-1}. The samples were prepared as Nujol mulls between CaF$_2$ plates under dry argon atmosphere.

4.2. X-ray structure determination

Data were collected on a Bruker SMART 1 K CCD diffractometer (λ(Mo Kα)-radiation, graphite monochromator, ω and φ scan mode) and corrected for absorption using the SADABS program [64]. For details, see Table S3. The structures were solved by direct methods and refined by full-matrix least squares technique on F^2 with anisotropic displacement parameters for non-hydrogen

atoms. Fluorine atoms of one trifluoromethyl group of one of the two independent molecules in **6** are disordered over two sites with the equal occupancies. The hydrogen atoms in both compounds were placed in calculated positions and refined within the riding model with fixed isotropic displacement parameters (U$_{iso}$-(H) = 1.5U$_{eq}$(C) for the CH$_3$-groups and U$_{iso}$(H) = 1.2U$_{eq}$(C) for the other groups). All calculations were carried out using the SHELXTL program [65].

4.3. Sn(acac)OSO$_2$CF$_3$ (**3**)

To a solution of tin(II) acetylacetonate (1.43 g, 4.44 mmol) in 10 ml of dichloromethane MeOTf (0.5 ml, 4.44 mmol) was added dropwise at room temperature. The reaction mixture was stirred for 2 h. The solvent was then removed under vacuum to about one fifth of the initial volume to give a crystalline precipitate. The precipitate was filtered, washed with cold chloroform, and dried in vacuo. Yield: 0.75 g (46%). Mp: 93–95 °C dec. Anal. Calc. for C$_6$H$_7$F$_3$O$_5$SSn (366.89 g): C, 19.64; H, 1.92. Found: C, 19.71; H, 1.90%. ^{1}H NMR (CDCl$_3$): 1.75 (s, 6H, CH$_3$), 5.64 (s, 1H, CH). ^{13}C NMR (CDCl$_3$): 21.0 (CH$_3$), 62.5 (CH), 140.9 (q, CF, $^1J_{CF}$ = 321.3 Hz), 176.1 (CO).

4.4. [Al[OCH(CF$_3$)$_2$]$_2$OCH$_2$CH$_2$NMe$_2$]$_2$ (**6**)

To a stirred solution of PhSn(OCH$_2$CH$_2$NMe$_2$)$_2$Cl (1.46 g, 3.58 mmol) in 10 ml of dichloromethane Ag[Al(OCH(CF$_3$)$_2$)$_4$] (2.86 g, 3.58 mmol) in 10 ml of dichloromethane was added dropwise. The reaction mixture was stirred for 2 h at room temperature and filtered through celite. The solvent was then removed under vacuum to about one fifth of the initial volume. A white crystalline precipitate was obtained. The precipitate was filtered, washed with cold chloroform, and dried in vacuo. Yield: 0.860 g (54%). Mp: 195–197 °C. Anal. Calc. for C$_{20}$H$_{24}$Al$_2$F$_{24}$N$_2$O$_6$ (898.34 g): C, 26.74; H, 2.69; N, 3.12. Found: C, 26.76; H, 2.66; N, 3.25%. ^{1}H NMR (CD$_2$Cl$_2$): 2.45 (s, 6H, NMe$_2$), 2.74 (t, 2H, CH$_2$N, ^3J = 5.9 Hz), 3.86 (t, 2H, CH$_2$O, ^3J = 5.9 Hz), 4.46 (sept, 2H, CH(CF$_3$)$_2$, $^3J_{HF}$ = 5.8 Hz). ^{13}C NMR (CD$_2$Cl$_2$): 45.1 (NMe$_2$), 57.6 (CH$_2$N), 61.9 (CH$_2$O), 71.2 (sept, CH(CF$_3$)$_2$, $^2J_{CF}$ = 32.9 Hz), 127.6 (q, CF$_3$, $^1J_{CF}$ = 286.0 Hz), 127.7 (q, CF$_3$, $^1J_{CF}$ = 286.0 Hz).

4.5. [Ph(C_6F_5)Sn(OCH$_2$CH$_2$NMe$_2$)$_2$][H$_2$OB(C_6F_5)$_3$] (**9**)

To a stirred solution of PhSn(OCH$_2$CH$_2$NMe$_2$)$_2$Cl (0.87 g, 2.13 mmol) in 20 ml of dichloromethane Ag[B(C_6F_5)$_4$] (1.68 g, 2.13 mmol) in 20 ml of dichloromethane was added. The reaction mixture was stirred overnight at room temperature and filtered through celite. Concentration to dryness under reduced pressure gave 2.02 g (88%) of off-white powder. Mp: 81–82 °C dec. Anal. Calc. for C$_{38}$H$_{27}$BF$_{20}$N$_2$O$_3$Sn (1051.11 g): C, 42.69; H, 2.55; N, 2.62. Found: C, 43.40; H, 2.50; N, 2.75%. ^{1}H NMR (CDCl$_3$): 2.52 (br. s, 12H, NMe$_2$), 2.82 (br. s, 4H, CH$_2$N), 3.97 (br. s, 4H, CH$_2$O), 7.35–7.48 (m, 3H, m- and p-Ph), 7.84 (s, 2H, o-Ph, $^3J_{SnH}$ = 50.8 Hz). ^{13}C NMR (CDCl$_3$): Me$_2$NCH$_2$CH$_2$O, 45.4 (br., 4C, NMe$_2$), 58.3 (s, 2C, CH$_2$N), 62.5 (br., 2C, CH$_2$O); B-C_6F_5, 123.1–124.7 (br., 3C, i-C),

136.2 (d, 6C, *m*-C, $^1J_{CF}$ = 246.0 Hz), 137.7 (d, 3C, *p*-C, $^1J_{CF}$ = 245.3 Hz), 148.1 (d, 6C, *o*-C, $^1J_{CF}$ = 239.9 Hz); Sn–C$_6$F$_5$, 136.6 (d, 2C, *m*-C, $^1J_{CF}$ = 268.6 Hz), 138.6 (d, 1C, *p*-C, $^1J_{CF}$ = 242.9 Hz), 147.8 (d, 2C, *o*-C, $^1J_{CF}$ = 235.4 Hz); Sn–Ph, 128.8 (s, 2C, *o*-Ph, $^2J_{SnC}$ = 113.2 Hz), 129.9 (s, 1C, *p*-Ph, $^4J_{SnC}$ = 22.0 Hz), 135.9 (s, 2C, *m*-Ph, $^3J_{SnC}$ = 58.8 Hz). ^{11}B NMR (CDCl$_3$): −16.85. ^{19}F NMR (CDCl$_3$): B-C$_6$F$_5$, −132.5 (d, 6F, *o*-F, J_{FF} = 9.18 Hz), −162.8 (t, 3F, *p*-F, J_{FF} = 20.65 Hz), −166.7 (t, 6F, *m*-F, J_{FF} = 18.4 Hz); Sn–C$_6$F$_5$, −135.5 (dd, 2F, *o*-F, $^3J_{SnF}$ = 293.7 Hz, J_{FF} = 21.8 Hz), −161.0 (dt, 1F, *p*-F, $^5J_{SnF}$ = 48.2 Hz, J_{FF} = 20.7 Hz), −165.3 (m, 2F, *m*-F). ^{119}Sn NMR (CDCl$_3$): −395.6. IR (Nujol, CaF$_2$, cm^{-1}): 3500–2000 (br).

Appendix A. Supplementary material

CCDC 718709 and 718710 contain the supplementary crystallographic data for **3** and **6**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2009.10.039.

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