Reverse Kocheshkov reaction – Redistribution reactions between RSn(OCH₂CH₂NMe₂)₂Cl (R = Alk, Ar) and PhSnCl₃: Experimental and DFT study

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ABSTRACT

A series of organotin compounds bearing two intramolecular N → Sn coordination bonds RSn(OCH₂CH₂N-Me₂)₂Cl (R = Me (**4**), n-Bu (**5**), Mes (**6**)) were synthesized in good yields. These compounds as well as **2** (R = Ph) react with PhSnCl₃ to give redistribution products RPhSnCl₂ and (Me₂NCH₂CH₂O)₂SnCl₂ (**3**). The direction of redistribution reactions is reverse to Kocheshkov reaction. DFT calculations have shown that the driving force of the reactions is formation of intramolecular N → Sn coordination bonds in (RO)₂SnCl₂ (**3**), the Lewis acid stronger than RSn(OR)₂Cl (**2**, **4–6**). The mechanism of the redistribution reaction between **2** and PhSnCl₃ consists of two steps: (1) initial exchange of OCH₂CH₂NMe₂ and Cl to give PhSn(OCH₂CH₂NMe₂)Cl₂ (**7**) followed by (2). Ph and OCH₂CH₂NMe₂ exchange.

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1. Introduction

One of the most widely used methods of synthesis of organotin compounds is the Kocheshkov reaction. Redistribution of hydrocarbon and halogen substituents at tin yields symmetrical organotin compounds at elevated temperatures (Scheme 1) [1].

Such order of redistribution of hydrocarbon substituents at tin atom takes place in all cases when tin is tetracoordinated. The increase of coordination number of tin due to interaction with electrodonating species (intermolecular complexes) or donor groups in side chains (intramolecular complexes) may lead to changes in activity of organotin compounds in respect to redistribution [2] or even change the direction of redistribution reactions.

It was shown with the use of multinuclear NMR spectroscopy that phenyltrihalogenestannanes PhSnHal₃ (Hal = Cl, Br) subjected to fluoride ions or tributylphosphane disproportionate in solution with the formation of Ph₂SnHal₂L$^{n-}_m$ and SnHal₄L$^{n-}_m$ (L = F, m = n = 1,2; L = PBu₃, m = 1, 2; n = 0) [3]. Later, the analogous disproportionation of monoorganotrichlorostannanes RSnCl₃ (R = Me, n-Bu, Ph) in presence of cis-1,2-bis(diphenylphosphino)-ethylene cis-Ph₂PCH=CHPPh₂ was observed in solution [4]. It was found that migration of phenyl group is faster than of aliphatic (Me and n-Bu) groups.

Disproportionation of organotin compounds bearing intramolecular coordination bonds was shown for (2,6-(Me₂NCH₂)C₆H₃)-SnPh₂X (**1**). These compounds (Scheme 2) are unstable in chloroform solution upon standing at room temperature. In two weeks the solution contains about 20% of disproportionation products [5]. Recently (Scheme 3) we have shown that interaction of hypercoordinated tin compound **2** with phenyltrichlorostannane leads to formation of dichlorodialkoxy derivative **3** and Ph₂SnCl₂ [6].

The above mentioned reactions are all reverse to Kocheshkov reaction because hydrocarbon and acceptor radicals are collected at different tin atoms. Since the data on this type of reactions are fragmentary, we performed more detailed experimental and theoretical investigation of reactions of **2** and its analogs RSn(OCH₂-CH₂NMe₂)₂Cl (R = Me (**4**), n-Bu (**5**), Mes (**6**)) with PhSnCl₃. The scope of the substituents R under study was dictated by experimental data on relative migration ability of hydrocarbon groups in Kocheshkov reaction: n-Bu < Me < Ph < Mes. Higher alkyl substituents, such as n-Bu, are the most reluctant to migrate, while bulky aryl groups (Mes) are the most active in redistribution reactions [1,7].
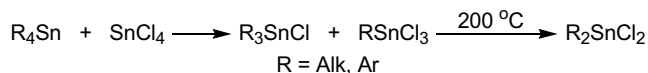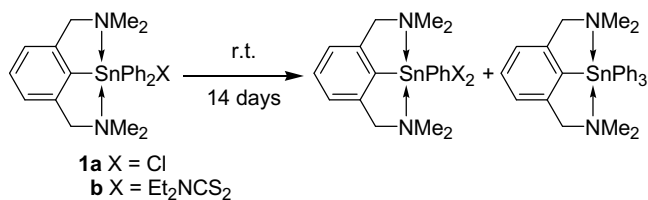
2. Results and discussion

2.1. Synthesis

Compounds **4–6** were obtained in good yields by the reaction of monoorganotin trichlorides with 2 equiv. of β-(N,N,-dimethyl)-aminoethoxytriethylstannane (Scheme 4).

R₄Sn + SnCl₄ ⟶ R₃SnCl + RSnCl₃ →(200 °C) R₂SnCl₂

R = Alk, Ar

Scheme 1.

1a X = Cl
b X = Et₂NCS₂

Scheme 2.

2　　+ PhSnCl₃ ⟶ **3**　　+ Ph₂SnCl₂

Scheme 3.

2 Et₃SnOCH₂CH₂NMe₂ + RSnCl₃ →(THF, reflux, 30 min) [structure] + 2 Et₃SnCl

4, R = Me; **5**, R = n-Bu; **6**, R = Mes
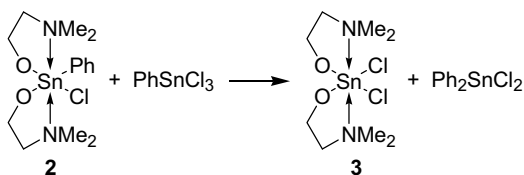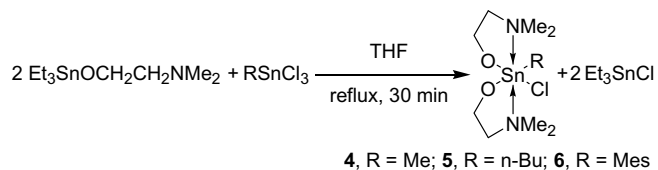
Scheme 4.

Compounds **2**, **4**–**6** were reacted with PhSnCl₃. Equimolar amounts of RSn(OCH₂CH₂NMe₂)₂Cl and PhSnCl₃ were mixed in THF solution and refluxed for 30 min. After solvent evaporation in vacuo solid residues were collected and analyzed using ¹H and

¹³C NMR. According to NMR data, the main products of the reactions are **3** [6] and RPhSnCl₂ (R = Ph [8], Me [9], Bu [10], Mes [11]). The yields are 76–89%. Small amounts of starting materials were also found in solution. Thus it was found that in presence of two dimethylaminoethoxy groups both alkyl and aryl radicals undergo redistribution under mild conditions.

2.2. DFT thermodynamics

To elucidate the reasons why the reverse Kocheshkov reactions take place we performed DFT study of the reactions of **2**, **4**–**6** with PhSnCl₃. In all cases the exchange of R and Cl groups is energetically favorable. Calculated Gibbs free energies ΔG–1 lie in a range from −7.4 to −22.6 kcal/mol (Scheme 5, Table 1). Contrary, if all N → Sn coordination bonds are broken in RSn(OCH₂CH₂NMe₂)₂Cl (**2**, **4**–**6**) and (Me₂NCH₂CH₂O)₂SnCl₂ (**3**), calculated Gibbs free energies ΔG–2 are positive, 1.4–8.1 kcal/mol.

It was found that the breaking of N → Sn coordination bonds in RSn(OCH₂CH₂NMe₂)₂Cl, except mesityl derivative **6**, is unfavorable. But the energy of breaking of N → Sn bonds in **3** is higher, 22.1 kcal/mol. Thus the calculations clearly show that the driving force of R and Cl exchange is the formation of intramolecular N → Sn coordination bonds in (RO)₂SnCl₂ (**3**), the Lewis acid stronger than RSn(OR)₂Cl (**2**, **4**–**6**). Indeed, according to thermodynamic cycle in Scheme 5, the energy difference between two reactions equals the difference between energies of breaking N → Sn bonds: $\Delta\Delta G = \Delta G\text{-}1 - \Delta G\text{-}2 = -22.1 - \Delta G\text{-}C$.

2.3. DFT mechanism

The mechanism of substituent redistribution reaction was studied for interaction of PhSn(OCH₂CH₂NMe₂)₂Cl (**2**) and PhSnCl₃ (Fig. 1 and Table S1). It was found that direct exchange of Ph and Cl groups is associated with high lying transition state TS-B (ΔG(TS-B) = 40.0 kcal/mol). In this transition state one of N → Sn coordination bond in **2** is broken. Ph group from **2** and Cl atom from PhSnCl₃ are bridging between two tin atoms (Fig. S1).

The alternative way consists of two steps: (1) initial exchange of OCH₂CH₂NMe₂ group from **2** and Cl atom from PhSnCl₃ with the formation two molecules of PhSn(OCH₂CH₂NMe₂)Cl₂ (**7**); (2) disproportionation of **7** via Ph and OCH₂CH₂NMe₂ exchange. The initial step is slightly energetically favorable, ΔG(**7**) = −1.9 kcal/mol.
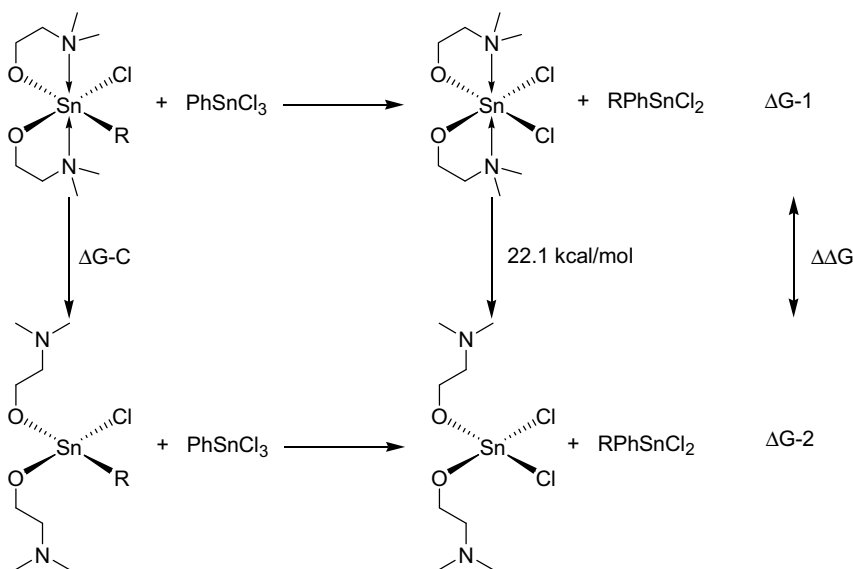
Scheme 5.

Table 1
Free energies of the reactions in Scheme 5 (kcal/mol)

R	ΔG-C	ΔG-1	ΔG-2	$\Delta\Delta G$
Me	8.0	−9.4	3.7	−13.1
Bu	7.6	−10.9	2.5	−13.5
Ph	12.3	−7.4	1.4	−8.7
Mes	−9.6	−22.6	8.1	−30.7

The potential energy surface along the reaction pathway is very flat. Thus we were unable to localize the corresponding transition state. In other words, **7** is formed from **2** and $PhSnCl_3$ with virtually no barrier.

The next step, Ph and $OCH_2CH_2NMe_2$ exchange between two molecules of **7**, goes via transition state TS-A (ΔG(TS-A) = 24.8 kcal/mol; Fig. S1). In TS-A both tin atoms are hexacoordinated and each has N → Sn coordination bond. There are a number of coordination isomers of TS-A possible. We performed optimization of more than 20 possible structures. The one presented is the lowest in energy.

Transition state TS-A is 15.2 kcal/mol lower in energy than TS-B. Thus we propose that the ligand exchange reaction goes solely via two-step mechanism with initial formation of **7** and further exchange of Ph and $OCH_2CH_2NMe_2$ groups. Moreover the calculated barrier of the reaction (24.8 kcal/mol) is consistent with the experimental result: the reaction proceeds to completion in 30 min. in refluxing THF (∼66 °C). We also suppose that exchange reactions between **4** and **6** and $PhSnCl_3$ proceed via the same mechanism.

3. Conclusion

We have shown that the presence of intramolecularly coordinating groups $OCH_2CH_2NMe_2$ at tin atom dramatically increase the mobility of substituents. Both higher alkyl as well as aryl groups in $RSn(OCH_2CH_2NMe_2)Cl_2$ readily redistribute in refluxing THF, while the exchange reaction between Ph_3SnCl and $PhSnCl_3$ yielding Ph_2SnCl_2 demands heating up to 200 °C.

The presence of donor groups lead to desymmetrization of tin compounds. Tin species bearing coordinating groups collect electronegative substituents and release electropositive ones. This is due to more effective coordination of donor groups in SnX_4, the Lewis acids stronger than $RSnX_3$ or R_2SnX_2.

We suppose that the activation of exchange reactions in mild conditions with the use of intramolecularly coordinating groups may lead to development of new methods of synthesis of organotin

Fig. 1. Potential energy surface of the reaction of **2** with $PhSnCl_3$ (Gibbs free energies in kcal/mol).

compounds bearing different aryl groups $ArAr'SnX_2$, which were not readily accessible previously.

4. Experimental

4.1. General procedures

All manipulations were carried out under purified argon using standard Schlenk and high-vacuum-line techniques. The commercially available solvents were purified by conventional methods and distilled immediately prior to use. $PhSnCl_3$ [7], $MeSnCl_3$ [7], $BuSnCl_3$ [7], $MesSnCl_3$ [11] and $Et_3Sn(OCH_2CH_2NMe_2)$ [12] were synthesized as described earlier. NMR spectra were recorded on an AVANCE-400 NMR spectrometer at 400.13 MHz (^1H) and 100.62 MHz (^{13}C) in THF-d_8. Chemical shifts are indirectly referenced to tetramethylsilane ($SiMe_4$) via the solvent signals. The accuracy of chemical shift measurements is ±0.01 ppm (^1H) and ±0.05 ppm (^{13}C). Elemental analyses were performed on a Carlo Erba EA1108 CHNS-O elemental analyzer.

4.2. $MeSn(OCH_2CH_2NMe_2)_2Cl$ (**4**)

A solution of $Et_3Sn(OCH_2CH_2NMe_2)$ (6.65 g, 23.33 mmol) in Et_2O (20 mL) was added dropwise to a stirred solution of $MeSnCl_3$ (2.80 g, 11.66 mmol) in Et_2O (30 mL). After 2 h of stirring an abundant white precipitate has formed. This was filtered, washed with hexane, and dried in vacuo. The yield was 2.49 g (61.8%). M.p. 139–140 °C. ^1H NMR (400.1 MHz, CD_2Cl_2, ppm): δ = 0.71 (br, s, $^2J_{SnH}$ = 99.0/103.6 Hz, 3 H, MeSn), 2.57 (br, s, 12 H, Me_2N), 2.70 (br, 4H, CH_2N), 3.78 (br, m, 4 H, CH_2O). ^{13}C NMR (100.61 MHz, CD_2Cl_2, ppm): δ = 10.80 (MeSn), 46.27 (Me_2N), 58.74 (br, CH_2N), 63.61 (br, CH_2O). Anal. calc. for $C_9H_{23}ClN_2O_2Sn$ (345.45): C, 31.29; H, 6.71; N, 8.11. Found: C, 31.05; H, 6.80; N, 7.97%.

4.3. $BuSn(OCH_2CH_2NMe_2)_2Cl$ (**5**)

A solution of $Et_3Sn(OCH_2CH_2NMe_2)$ (3.42 g, 11.63 mmol) in THF (25 mL) was added dropwise to a stirred solution of $BuSnCl_3$ (1.64 g, 5.82 mmol) in THF (30 mL). The mixture was stirred for 2 h and stored overnight. The solvent was removed in vacuo to about one sixth of the initial volume, then 50 mL of hexane was added to give a white crystalline precipitate. This was filtered under argon, washed with hexane, and dried in vacuo. The yield was 1.53 g (68%). ^1H NMR (400.1 MHz, $CDCl_3$, ppm): δ = 0.75–0.79 (m, 3H, $SnCH_2CH_2CH_2CH_3$), 1.17–1.25 (m, 2H, $SnCH_2CH_2CH_2CH_3$), 1.41–1.55 (m, 4H, $SnCH_2CH_2CH_2CH_3$), 2.58 (s, 12H, Me_2N), 2.64–2.69 (br, m, 4H, CH_2N), 3.36–3.81 (br, m, J_{SnH} = 66.3 Hz, 4H, CH_2O). ^{13}C NMR (100.61 MHz, $CDCl_3$, ppm): δ = 13.35 ($SnCH_2CH_2CH_2CH_3$), 26.16 ($SnCH_2CH_2CH_2CH_3$), 29.43 (J_{SnC} = 48.3 Hz), 30.53 ($SnCH_2CH_2CH_2CH_3$), 46.01 (Me_2N), 58.32 (CH_2N), 63.12 (CH_2O). Anal. Calc. for $C_{12}H_{29}ClN_2O_2Sn$ (387.51): C, 37.19; H, 7.54; N, 7.23. Found: C, 37.05; H, 7.76; N, 7.14%.

4.4. $MesSn(OCH_2CH_2NMe_2)_2Cl$ (**6**)

A solution of $MesSnCl_3$ (2.10 g, 6.10 mmol) in THF (30 mL) was added dropwise to a stirred solution of $Et_3Sn(OCH_2CH_2NMe_2)$ (3.95 g, 13.43 mmol) in THF (25 mL). The mixture was stirred for 2 h and stored overnight. The solvent was removed in vacuo to about one sixth of the initial volume, and then 50 mL of hexane was added to give a white crystalline precipitate. This was filtered under argon, washed with hexane, and dried in vacuo. The yield was 1.78 g (65%). ^1H NMR (400.1 MHz, $CDCl_3$, ppm): 2.18 (s, J_{SnH} = 8.8 Hz, 3H, p-CH_3), 2.22 (br, s, 12H, Me_2N), 2.47 (s, J_{SnH} = 5.9 Hz, 6H, o-CH_3), 2.66 (br, m, 4H, CH_2N), 4.00 (br, t,
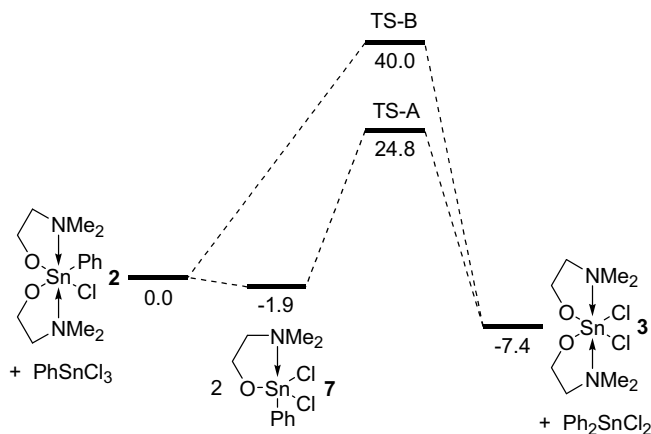
J_{HH} = 5.4 Hz, 4 H, CH$_2$O), 6.85 (s, J_{SnH} = 226.3 Hz, 2H, aromatic protons in Mes). ^{13}C NMR (100.61 MHz, CDCl$_3$, ppm): δ = 20.79 (br, *p*-CH$_3$), 24.38 (J_{SnC} = 37.7 Hz, *o*-CH$_3$), 45.09 (br, Me$_2$N), 60.39 (br, CH$_2$N), 61.55 (CH$_2$O), 126.68 (C$_m$), 132.60 (C$_i$), 136.70 (C$_p$), 141.78 (C$_o$). Anal. Calc. for C$_{17}$H$_{31}$ClN$_2$O$_2$Sn (449.58): C, 45.42; H, 6.95; N, 6.23. Found: C, 45.35; H, 7.06; N, 6.12%.

4.5. General procedure for the reaction of RSn(OCH$_2$CH$_2$NMe$_2$)$_2$Cl with PhSnCl$_3$ (NMR experiment)

A solution of RSn(OCH$_2$CH$_2$NMe$_2$)$_2$Cl (0.6 mmol) and PhSnCl$_3$ (0.6 mmol) in THF (10 mL) was mixed. After 30 min in refluxing THF the solvent was removed in vacuo and CD$_2$Cl$_2$ (1 mL) was introduced. The solution was transferred to a 5-mm NMR tube and sealed.

4.6. Reaction with PhSn(OCH$_2$CH$_2$NMe$_2$)$_2$Cl (**2**)

According to the ^{1}H and ^{13}C NMR spectra, the solution contained (Me$_2$NCH$_2$CH$_2$O)$_2$SnCl$_2$ (**3**) and Ph$_2$SnCl$_2$ [8] (89%) as the main products. Ph$_2$SnCl$_2$: ^{1}H NMR (400.1 MHz, CD$_2$Cl$_2$, ppm): δ = 7.60–7.67 (m, 3 H, Ph), 7.81–7.84 (m, 2 H, Ph). ^{13}C NMR (100.61 MHz, CDCl$_3$, ppm): δ = 130.32 ($^3J_{SnC}$ = 85.1 Hz, C$_m$), 132.48 ($^4J_{SnC}$ = 18.4 Hz, C$_p$), 135.57 ($^2J_{SnC}$ = 63.6 Hz, C$_o$), 137.50 (C$_i$).

4.7. Reaction with MeSn(OCH$_2$CH$_2$NMe$_2$)$_2$Cl (**4**)

According to the ^{1}H and ^{13}C NMR spectra, the solution contained (Me$_2$NCH$_2$CH$_2$O)$_2$SnCl$_2$ (**3**) and MePhSnCl$_2$ [9] (85%) as the main products. MePhSnCl$_2$: ^{1}H NMR (400.1 MHz, CD$_2$Cl$_2$, ppm): δ = 1.35 (s, CH$_3$), 7.52–7.68 (m, 5 H, Ph). ^{13}C NMR (100.61 MHz, CDCl$_3$, ppm): δ = 4.80 (CH$_3$), 129.6 (C$_m$), 131.7 (C$_p$), 134.6 (C$_o$), 138.8 (C$_i$).

4.8. Reaction with BuSn(OCH$_2$CH$_2$NMe$_2$)$_2$Cl (**5**)

According to the ^{1}H and ^{13}C NMR spectra, the solution contained (Me$_2$NCH$_2$CH$_2$O)$_2$SnCl$_2$ (**3**) and BuPhSnCl$_2$ [10] (76%) as the main products. BuPhSnCl$_2$: ^{1}H NMR (400.1 MHz, CD$_2$Cl$_2$, ppm): δ = 0.85–0.91 (m, 3H, SnCH$_2$CH$_2$CH$_2$CH$_3$), 1.30–1.36 (m, 2H, SnCH$_2$CH$_2$CH$_2$CH$_3$), 1.45–1.62 (m, 4H, SnCH$_2$CH$_2$CH$_2$CH$_3$), 7.26–7.35 (m, 3 H, Ph), 7.60–7.67 (m, 2 H, Ph). ^{13}C NMR (100.61 MHz, CDCl$_3$, ppm): δ = 13.85 (SnCH$_2$CH$_2$CH$_2$CH$_3$), 25.24, 25.53 (SnCH$_2$CH$_2$CH$_2$CH$_3$), 27.94 (SnCH$_2$CH$_2$CH$_2$CH$_3$), 127.27 (C$_m$), 127.86 ($^4J_{SnC}$ = 21.5 Hz, C$_p$), 134.89 ($^2J_{SnC}$ = 66 Hz, C$_o$), 136.22 (C$_i$).

4.9. Reaction with MesSn(OCH$_2$CH$_2$NMe$_2$)$_2$Cl (**6**)

According to the ^{1}H and ^{13}C NMR spectra, the solution contained (Me$_2$NCH$_2$CH$_2$O)$_2$SnCl$_2$ (**3**) and MesPhSnCl$_2$ [11] (80%) as the main products. MesPhSnCl$_2$: ^{1}H NMR (400.1 MHz, CD$_2$Cl$_2$, ppm): δ = 2.29 (c, 3H, *p*-Me), 2.52 (s, J_{SnH} = 10 Hz, 6H, *o*-Me), 6.95 (c, J_{SnH} = 28 Hz, 2H, *m*-H-Mes), 7.51 (m, 3H, *m*-Ph, *p*-Ph), 7.76 (m, 2 H, *o*-Ph). ^{13}C NMR (100.61 MHz, CDCl$_3$, ppm): δ = 21.1 (J_{SnC} = 9 Hz, *p*-Me), 25.3 (J_{SnC} = 48 Hz, *o*-Me), 129.4 (J_{SnC} = 79/82 Hz, *m*-Mes), 129.6 (J_{SnC} = 81/84 Hz, *m*-Ph), 131.1 (J_{SnC} = 17 Hz, *p*-Ph), 134.3 (J_{SnC} = 62/64 Hz, *o*-Ph), 135.2 (*i*-Ph), 142.1 (J_{SnC} = 15 Hz, *p*-Mes), 142.6 (*i*-Mes), 144.2 (J_{SnC} = 60/62 Hz, *o*-Mes).

5. Computational method

All calculations were done at the DFT level of theory. The geometry optimizations were carried out using the PBE generalized gra-

dient functional [13]. The triple zeta valence basis set including polarization functions TZ2P (3,1,1/3,1,1/1,1) for Sn, Cl, C, N, O atoms and (3,1,1/1) for H atom was used [14]. Innermost electrons for Sn, Cl, C, N, O atoms were treated using the ECP-SBKJC relativistic effective core potentials [15].

Total energies E, zero point vibration energies ZPE, $E^0 = E$ + ZPE, H^0, G^0 were calculated for all stationary points corresponding to reactants, transition states, intermediates, and products. Vibrational frequencies were used to characterize stationary points either as minima (number of imaginary frequencies i = 0) or transition states (i = 1). For reliable identification of transition states, the IRC procedure, following the reaction path from transition state to products/reactants, was used [16]. All calculations were performed using the PRIRODA program [17].

Appendix A. Supplementary material

Calculated structures, energetic parameters. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2008.09.043.

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