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Palladium^{II} Complexes of the (N,C,N)SnCl Stannylene

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*Summary: The reactions of heteroleptic stannylene (N,C,N)SnCl (**1**), where N,C,N is an abbreviation for N,C,N-chelate 2,6-(Me_2NCH_2)$_2C_6H_3$, with selected palladium complexes resulted in new Sn^{II}−Pd^{II} complexes in which **1** itself acts as a 2e-donor ligand, resulting in a Cl−Pd^{II}−Sn^{II}−Cl structural motif, instead of a Pd^{II}−Sn^{II}−Cl_2, which would be expected in the case of insertion of **1** into a Pd^{II}−Cl bond.*

The chemistry of transition metal (TM)−Sn^{II} complexes has been developed into an active area of research during the last two decades, as they are believed to be preformed catalysts for several reactions depending on the type of TM.[1] The most common way to prepare such complexes is the reaction of a TM−Cl complex with a stannylene SnR_2 (where R is an organic or inorganic group). In such reactions SnR_2 compounds may behave as a two-electron ligand,[2] a substrate for insertion into a TM−Cl bond,[3] or a reducing agent.[4] Although examples where Sn^{II} compounds behave as two-electron σ donors (tertiary phosphine analogues) are known, only a limited number of such reactions have been reported so far.[5] For this reason, the study of the chemistry of TM− Sn^{II} compounds with side-chain substituents bearing nitrogen[6] and oxygen[7] donors, so-called C,Y-chelates, at the tin(II) atom may be of interest. In such functionalized compounds we can expect an increase in the Lewis base character due to donation of electron density to the Sn^{II} center by a donor heteroatom. As a result, a greater ability to form complexes with Lewis acids such as transition metal compounds can be expected.

In this class of compounds, halostannylenes are of particular utility for the synthesis of many functionalized stannylenes, but their preparation can be rather problematic in itself. Introduction of Y,C,Y-chelates into a Sn^{II} fragment, however, has been shown to be an efficient method of preparation of stable halostannylenes. Interestingly, despite the relatively long history of organostannylene (N,C,N)SnCl (**1**),[6c] investigation of its reactivity is limited to several reactions, in particular to oxidative additions.[6b,c]

Here we report the preparation of ((N,C,N)SnCl)(2-(Me_2NCH_2)C_6H_4)PdCl (**2**)[8] and ((N,C,N)SnCl)$_2$PdCl_2 (**3**)[9] (Scheme 1) by the reactions of **1** with selected complexes of Pd, namely, ({2-(Me_2NCH_2)C_6H_4}Pd-μ-Cl)$_2$ and (PPh_3)$_2$PdCl_2.

Compound **2** was prepared by the reaction of 2 equiv of **1** with the dimeric Pd^{II} complex bis(μ-chloro-2-(dimethylami-

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(8) Synthesis of {2,6-(Me_2NCH_2)$_2C_6H_3$SnCl}{2-(Me_2NCH_2)C_6H_4}PdCl (**2**). A solution of (NCN)SnCl (345 mg, 1 mmol) in THF (40 mL) was stirred with ({2-(Me_2NCH_2)C_6H_4}Pd-μ-(Cl))$_2$ (276 mg, 0.5 mmol) for 1 day. Solvent was evaporated, and the solid residue washed with pentane (2 × 5 mL) to give yellow solid **2** (yield 610 mg, 98 %). For **2**: mp 186 °C dec. Anal. Calcd for $C_{21}H_{31}N_3Cl_2SnPd$ (621.49): C, 40.58; H, 5.03. Found: C, 40.85; H, 5.15. ^{1}H NMR (C_6D_6, 500,13 MHz): δ (ppm) 2.23 (s, 6H, NCH_3), 2.54 (s, 6H, NCH_3), 2.60 (s, 6H, NCH_3), 3.35 (AX system, 2H, CH_2N), 3.55 (s, 2H, CH_2N), 3.77 (AX system, 2H, CH_2N), 6.85 (m, 2H, ArH), 6.92 (d, 2H, ArH), 7.02 (t, 1H, ArH), 7.2 (m, 1H, ArH), 8.53 (d, 1H, ArH). ^{13}C NMR (C_6D_6, 125.77 MHz): δ (ppm) 46.5 (NCH_3), 47.2 (NCH_3), 50.2 (NCH_3), 64.3 (CH_2N), 72.6 (CH_2N), 121.9, 122.9, 126.5, 128.2, 130.2, 140.2, 142.8, 144.1, 147.8, 150.2. ^{119}Sn NMR (C_6D_6, 186.49 MHz): δ (ppm) −76.5.

(9) Synthesis of {2,6-(Me_2NCH_2)$_2C_6H_3$SnCl}$_2$PdCl_2 (**3**). A solution of (NCN)SnCl (1.1 g, 3 mmol) in THF (40 mL) was stirred with (PPh_3)$_2$-PdCl_2 (702 mg, 1 mmol) 1 day. The resulting mixture was filtrated, and the yellow solid residue was washed with toluene (2 × 15 mL) to give yellow solid **3** (yield 782 mg, 90 %). For **3**: mp 198 °C dec. Anal. Calcd for $C_{24}H_{38}N_4Cl_4Sn_2Pd$ (868.19): C, 33.20; H, 4.41. Found: C, 33.50; H, 4.55. ^{1}H NMR ($CDCl_3$, 500.13 MHz): δ (ppm) 2.63 (s, 6H, NCH_3), 2.87 (s, 6H, NCH_3), 3.70 (AX system, 2H, CH_2N), 4.15 (AX system, 2H, CH_2N), 6.99 (d, 2H, ArH), 7.32 (t, 1H, ArH). ^{13}C NMR ($CDCl_3$, 125.77 MHz): δ (ppm) 47.0 (NCH_3), 47.1 (NCH_3), 63.7 (CH_2N), 125.3, 130.2, 141.9, 145.3. ^{119}Sn NMR ($CDCl_3$, 186.49 MHz): δ (ppm) −122.2.

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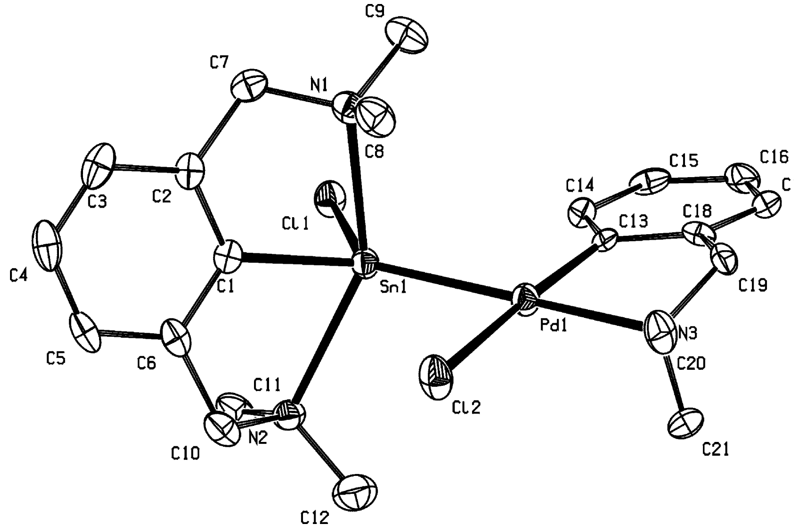
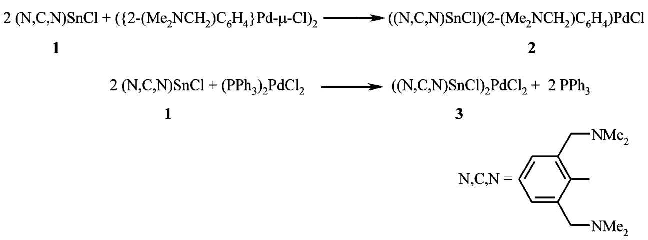


Figure 1. ORTEP view of **2**. The thermal ellipsoids are drawn with 50% probability. Hydrogen atoms are omitted for clarity. Selected bond distances (Å) and angles (deg): Sn(1)−C(1) 2.117(6), Sn(1)−N(1) 2.424(5), Sn(1)−Cl(1) 2.431(2), Sn(1)−Pd(1) 2.4956(8), Sn(1)−N(2) 2.504(5), Pd(1)−C(13) 1.999(6), Pd(1)−N(3) 2.154(4), Pd(1)−Cl(2) 2.398(2), C(1)−Sn(1)−N(1), 74.3(2), C(1)−Sn(1)−Cl(1) 97.19(15), N(1)−Sn(1)−Cl(1) 89.12(12), C(1)−Sn(1)−Pd(1) 138.18(14), N(1)−Sn(1)−Pd(1) 98.75(11), Cl(1)−Sn(1)−Pd(1) 124.26(4), C(1)−Sn(1)−N(2) 73.7(2), N(1)−Sn(1)−N(2) 147.84(15), Cl(1)−Sn(1)−N(2) 91.46(12), Pd(1)−Sn(1)−N(2) 107.29(11).

Scheme 1. Schematic Formation of Complexes 2 and 3



nomethyl)phenylpalladium). The ^{1}H and ^{13}C NMR spectra of **2** indicate 1:1 molar stoichiometry of organic groups bound at Sn^{II} and Pd^{II} atoms. The spectra suggest C_s symmetry of the complex in solution, with asymmetric CH$_a$H$_b$NMe$_a$Me$_b$ moieties of the N,C,N substituent and a symmetric CH$_2$NMe$_2$ moiety of the 2-(Me$_2$NCH$_2$)C$_6$H$_4$ ligand. While the three equally intense NMe signals have not been assigned to a specific ligand, the CH$_a$H$_b$ protons give rise to an AX spin system. The ^{119}Sn NMR signal of **2** is at $\delta(^{119}\mathrm{Sn})$ −76.5, corresponding to an upfield shift of 227 ppm from the starting **1**. These spectral features, however, do not clarify whether **1** itself acts as a 2*e*-donor ligand via the Sn atom or whether an insertion into the Pd−Cl bond has taken place.[10] Therefore, the crystal structure of **2** was determined by X-ray diffraction techniques. The molecular structure of **2**[11] (Figure 1) clearly shows that **1** acts as a 2*e*-donor ligand, resulting in a Cl−Pd^{II}−Sn^{II}−Cl structural motif, instead of a Pd^{II}−Sn^{II}−Cl$_2$, which would be expected in the case of insertion of **1** into a Pd^{II}−Cl bond.

The square-planar geometry of the central Pd^{II} atom (Σ = 360.1(3)°) is typical for d^8 metal ions, and the largest deformation could be found in the angle Sn(1)−Pd(1)−C(13), 102.05(15)°. The value of the Pd(1)−N(3) bond length (2.154(4) Å) indicates the presence of a strong interaction with a d^8 metal and is comparable to those found in similar monomeric Pd complexes containing similar C,N-chelating ligands (range of 2.140−2.155 Å),[12] but is longer than those found in the starting dimeric compound ({2-(Me$_2$NCH$_2$)C$_6$H$_4$}Pd-μ-Cl)$_2$ (2.073 Å).[13] The Sn^{II} atom is coordinated *trans* to the nitrogen atom N(3) (angle Sn(1)−Pd(1)−N(3) = 174.60(13)°). The coordination environment of the Sn(1) atom can be best described as a *trans*-trigonal bipyramid in which Pd(1), C(1), and Cl(1) atoms form an equatorial plane (Σ = 360.1(3)°) and both nitrogen atoms are in axial positions. Most interestingly, the value of the Pd(1)−Sn(1) bond distance, being 2.4956(8) Å, is shorter than any of the Pd^0−Sn^{II} distances in the Pd{Sn(N(SiMe$_3$)$_2$)$_2$}$_3$ (2.533(1), 2.540(1), 2.517(1) Å) and comparable to that found in another Pd^0−Sn^{II} complex ({^{i}Pr$_2$PC$_2$H$_4$P^{i}Pr$_2$}Pd{Sn(CH(SiMe$_3$)$_2$)$_2$}), where a Pd^0−Sn^{II} distance of 2.481(2) Å indicated a Pd═Sn double bond. Furthermore the relatively small C(1)−Sn(1)−Cl(1) angle of 97.19(16)° is less than that in an ideal trigonal bipyramidal or even in a tetrahedral arrangement of the Sn atoms and is smaller than mean C−Sn−C and N−Sn−N angles found in {^{i}Pr$_2$PC$_2$H$_4$P^{i}Pr$_2$}Pd{Sn(CH(SiMe$_3$)$_2$)$_2$} (98.8(8)°) and Pd{Sn(N(SiMe$_3$)$_2$)$_2$}$_3$ (107(1)°). The values of the Sn(1)−N(1) and Sn(1)−N(2) bonding distances, 2.424(5) and 2.505(5) Å, indicate the presence of a medium-strength Sn−N intramolecular interaction in **2**.

In a similar way, 2 equiv of **1** was stirred with (PPh$_3$)$_2$PdCl$_2$ in THF for 1 day to give a yellow suspension (see Scheme 1), and both fractions have been monitored with the help of ^{31}P and ^{119}Sn NMR spectroscopy. The THF solution showed one highly intense signal at −4.5 ppm and a very small one at 24.6

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[10] The ^{119}Sn NMR data of such complexes are very rare, and they are mostly missing.

[11] Yellow crystals of **2** were obtained by slow diffusion of pentane into a benzene solution of **2**. The intensity data were collected on a KUMA KM-4 CCD kappa-axis diffractometer using graphite-monochromated Mo Kα radiation (=0.71073 Å) at 260(2) K. C$_{21}$H$_{31}$Cl$_2$N$_3$PdSn, M = 621.48, monoclinic, space group $P2(1)/c$, a = 15.227(3) Å, b = 10.349(2) Å, c = 16.024(3) Å, α = 90°, β = 106.86(3)°, γ = 90°, V = 2416.5(8) Å^3, Z = 4, ρ = 1.708 g cm^{-3}, μ = 2.010 mm^{-1}, 19 053 reflections collected, of which 2814 were independent [R(int) = 0.0673]. Final R indices [$I > 2\sigma(I)$]: R1 = 0.0376, wR2 = 0.0651. The structure was solved by direct methods (SHELX-97,[15] SHELXTL V 5.1[16]). Non-hydrogen atoms were refined anisotropically, while hydrogen atoms were inserted in calculated positions and isotropically refined assuming a "riding" model.

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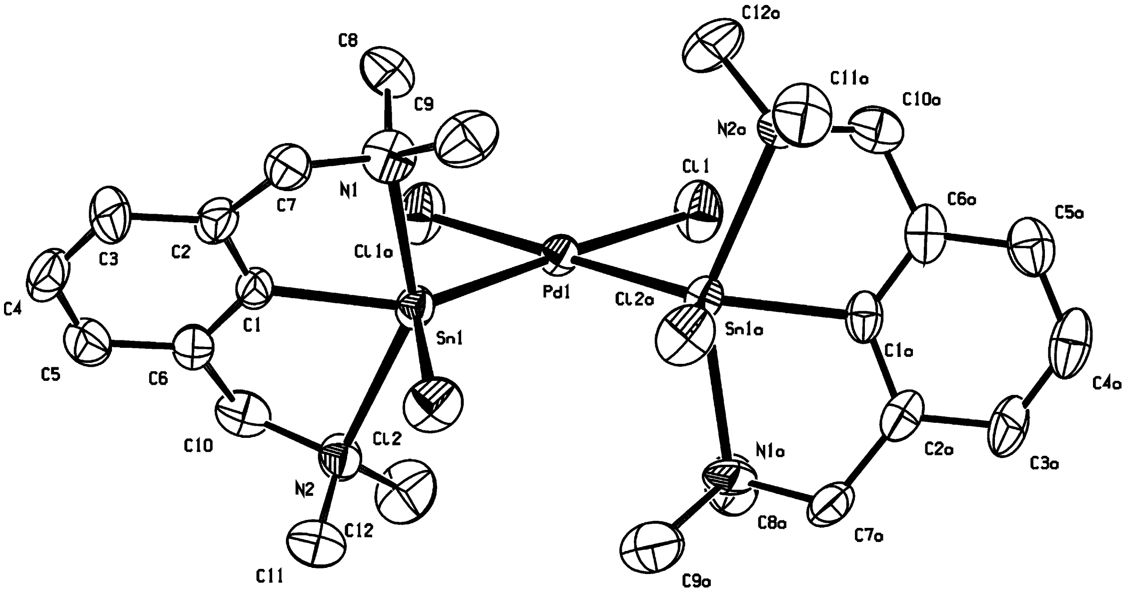

Figure 2. ORTEP view of **3**. The thermal ellipsoids are drawn with 50% probability. Hydrogen atoms are omitted for clarity. Selected bond distances (Å) and angles (deg): Sn(1)−C(1) 2.121(1), Sn(1)−Cl(2) 2.384(4), Sn(1)−N(1) 2.422(1), Sn(1)−N(2) 2.499(1), Sn(1)−Pd(1) 2.5197(9), Pd(1)−Cl(1) 2.360(3), Pd(1)−Cl(1a) 2.360(4), Pd(1)−Sn(1a) 2.5197(9), C(1)−Sn(1)−Cl(2) 97.2(4), C(1)−Sn(1)−N(1) 74.8(4), Cl(2)−Sn(1)−N(1) 89.5(3), C(1)−Sn(1)−N(2) 74.0(4), Cl(2)−Sn(1)−N(2) 90.7(3), N(1)−Sn(1)−N(2) 148.6(4), C(1)−Sn(1)−Pd(1) 137.9(4), Cl(2)−Sn(1)−Pd(1) 124.89(10), N(1)−Sn(1)−Pd(1) 102.3(3), N(2)−Sn(1)−Pd(1) 103.3(3), Cl(1)−Pd(1)−Cl(1a) 101.41(18), Cl(1a)−Pd(1)−Sn(1) 80.40(9), Cl(1)−Pd(1)−Sn(1) 177.85(10), Sn(1)−Pd(1)−Sn(1a) 97.80(4).

ppm assigned to free PPh_3 and the starting $(PPh_3)_2PdCl_2$ in the ^{31}P NMR spectrum, and no signal was observed in the ^{119}Sn NMR spectrum. These observations indicate that the whole amount of the starting **1** was consumed during the reaction with $(PPh_3)_2PdCl_2$, replacing PPh_3 groups that has been detected as free noncoordinated species in the reaction mixture. Solid material (insoluble in THF) was dissolved in $CDCl_3$, and a new signal at −122.2 ppm was detected in the ^{119}Sn NMR spectrum, indicating the formation of a new tin-containing compound that was characterized as $((N,C,N)SnCl)_2PdCl_2$ (**3**). The ^1H NMR spectrum of **3** also showed the presence of asymmetric CH_aH_b-NMe_aMe_b moieties of the N,C,N substituent (the CH_aH_b protons give rise to an AX spin system and two equally intense NMe signals), while the ^{31}P NMR spectrum of **3** showed no signal, indicating that both PPh_3 groups of the starting $(PPh_3)_2PdCl_2$ complex were replaced by **1**. It was also found that the use of an excess of **1** ($Sn^{II}/Pd^{II} = 3/1$) gives a better yield.

The crystal structure of **3** was determined by X-ray diffraction techniques and is depicted in Figure 2.[14] **1** again behaves as a tertiary phosphine analogue, resulting in the $Cl_2Pd^{II}{-}Sn^{II}Cl$ motif similar to **2**. The square-planar geometry of the central Pd^{II} atom ($\sum = 359.6(10)°$) is again retained in **3**, and while the values of both angles Cl(1a)−Pd(1)−Sn(1) and Cl(1a)−Pd(1)−Sn(1a) are the same (80.40(9)°), the largest deformation can be found in Cl(1)−Pd(1)−Cl(1a), 101.41(18)°. Interestingly, both the Sn(1) and Sn(1a) atoms have exactly the same coordination environment, having the same values of bond distances and bonding angles. This coordination geometry of both Sn atoms can best be described as a *trans*-trigonal bipyramid in which Pd(1), C(1), and Cl(2) atoms form an equatorial plane ($\sum = 359.9(6)°$) and both nitrogen atoms are in axial positions, similar to **2**. The value of the Sn(1)−Pd(1) bond distance, being 2.5197(9) Å, is again comparable to those found in the $Pd^0{-}Sn^{II}$ complex $Pd\{Sn(N(SiMe_3)_2)_2\}_3$ (2.533(1), 2.540(1), 2.517(1) Å) and is somewhat longer than that found in **2** and $\{^iPr_2PC_2H_4P^iPr_2\}Pd\{Sn(CH(SiMe_3)_2)_2\}$.

In summary, it was shown that heteroleptic stannylene (N,C,N)SnCl (**1**) can act as a 2*e*-donor ligand in selected Pd^{II} complexes resulting in a $Cl{-}Pd^{II}{-}Sn^{II}{-}Cl$ structural motif.

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Supporting Information Available: Crystallographic data for compounds **2** and **3** have been deposited with the Cambridge Crystallographic Data Centre, CCDC nos. 637856 and 639683. Further details of the structure determination of compounds **2** and **3**, including atomic coordinates, anisotropic displacement parameters, and geometric data (.cif files) are available free of charge via the Internet at http://pubs.acs.org.

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(14) Yellow crystals of **3** were obtained by slow evaporation of a CH_2Cl_2 solution of **3**. The intensity data for a single crystal of **3** were measured on a four-circle KappaCCD diffractometer with CCD area detector by monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) at 150(2) K. $C_{24}H_{38}Cl_4N_4Pd$, $M = 868.16$, tetragonal, space group $P4_12_12$, $a = 9.0340(7)$ Å, $b = 9.0340(11)$ Å, $c = 38.189(4)$ Å, $\alpha = 90°$, $\beta = 90°$, $\gamma = 90°$, $V = 3116.7(6)$ Å^3, $Z = 4$, $\rho = 1.850$ g cm^{-3}, $\mu = 2.524$ mm^{-1}, 10 441 reflections collected, of which 2272 were independent [R(int) = 0.0725]. Final R indices [$I > 2\sigma(I)$]: R1 = 0.0657, wR2 = 0.1418. The structure was solved by the direct methods (SIR97) and refined by a full-matrix least-squares procedure based on F^2 (SHELXL97). Hydrogen atoms were mostly localized on a difference Fourier map; however to ensure uniformity of treatment of the crystal, all hydrogens were recalculated into idealized positions (riding model) and assigned temperature factors $H_{iso}(H) = 1.2U_{eq}$(pivot atom) or $1.5U_{eq}$ for the methyl moiety.

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