Organometallics **1989,** *8,* 1373–1375

compound **2a**, including experimental details and crystal data and tables of positional and thermal parameters, general temperature factor expressions, bond distances and angles, torsion angles, intermolecular contacts up to 3.60 Å, and intensity data (27 pages); a listing of observed and calculated structure factors (20 pages) ordering information is given on any current masthead page.

Synthesis and Characterization of {2,6-Bis[(dimethylamino)methyl]phenyl}tin(II) Chloride and {2,6-Bis[(dimethylamino)methyl]phenyl}4-tolyltin(II), the First Example of a Mixed Diaryltin(II) Compound

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Summary: The bis ortho-chelated aryltin(II) chloride $\mathrm{Sn^{II}Cl[C_6H_3(CH_2NMe_2)_2\text{-}2,6]}$ (**1**) has been synthesized which has both in the solid state (X-ray) and in solution (^{1}H, ^{13}C, and ^{119}Sn NMR) a Ψ-trigonal-bipyramidal structure. Reaction of **1** with 4-tolyllithium gives the first example of a mixed diaryltin(II) compound, $\mathrm{Sn^{II}[C_6H_3(CH_2NMe_2)_2\text{-}2,6](C_6H_4Me\text{-}4)}$ (**2**), which readily undergoes oxidative addition reactions with electrophiles MeI and I_2.

Previously we have shown that the, potentially tridentate, monoanionic 2,6-bis[(dimethylamino)methyl]phenyl group, i.e. $\mathrm{[C_6H_3(CH_2NMe_2)_2\text{-}2,6]}$, may be used to prepare organotransition-metal compounds in which the metal has an unusual oxidation state for an organometallic compound, e.g. Fe(III)[1], Ni(III),[2] and Co(II).[3] Moreover, this aryl ligand allows the isolation of reactive intermediates; for example, {2,6-bis[(dimethylamino)methyl]phenyl}platinum iodide reacts with I_2 to give a complex in which I_2 is end-on coordinated to the platinum atom.[4]

We have extended our work with $\mathrm{[C_6H_3(CH_2NMe_2)_2\text{-}2,6]}$ to the preparation of divalent organotin compounds. Although a Mössbauer spectroscopic study of $\mathrm{SnCl[C_6H_3(CH_2NMe_2)_2\text{-}2,6]}$ and some related species that were assumed to be diaryltin(II) compounds has been published, this paper contains no further structural information.[5] This is unfortunate since {2,6-bis[(dimethylamino)methyl]phenyl}tin(II) compounds are of potential interest for two reasons. In the first place, intramolecular Sn–N coordination should stabilize the tin(II) state and this is likely to have interesting stereochemical consequences, and secondly, the synthesis of unknown mixed RR′Sn species might be accessible via RSnCl. We here report our first results in this area.

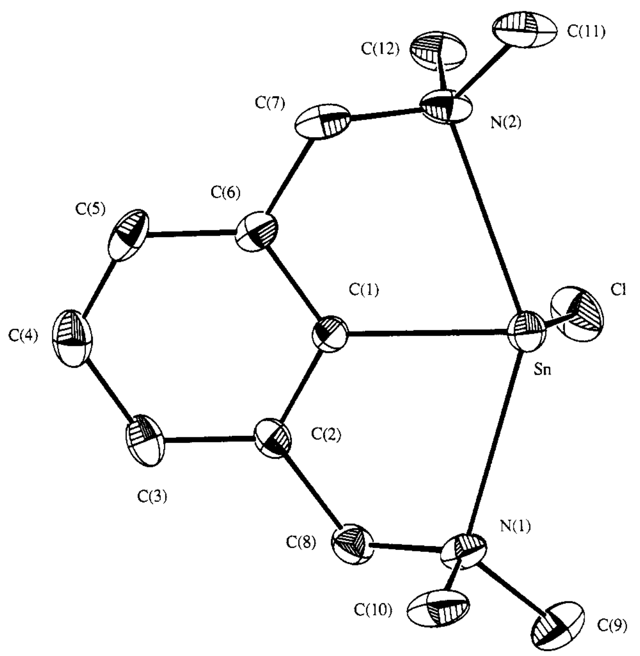
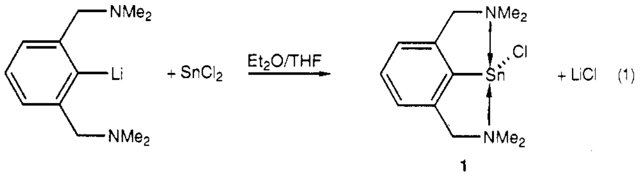


Figure 1. ORTEP drawing of 1. Bond distances (Å): Sn–C(1), 2.158 (8); Sn–Cl, 2.488 (3); Sn–N(1), 2.525 (8); Sn–N(2), 2.602 (8). Bond angles (deg): C(1)–Sn–Cl, 95.0 (3); C(1)–Sn–N(1), 71.9 (3); C(1)–Sn–N(2), 71.5 (3); Cl–Sn–N(1), 86.3 (2); Cl–Sn–N(2), 92.2 (3).

The reaction of $\mathrm{Li[C_6H_3(CH_2NMe_2)_2\text{-}2,6]}$ with $SnCl_2$, in a 1:1 molar ratio, in Et_2O/THF (eq 1), affords {2,6-bis[(dimethylamino)methyl]phenyl}tin chloride (1) which is obtained, after workup, as a white solid[6] in quantitative yield.



We have now fully characterized this interesting aryltin(II) chloride. Compound 1 is readily soluble in hydrocarbon and ether solvents, and molecular weight determinations[6] (cryoscopy in benzene) indicate that it is a monomer in solution. Single crystals of 1, suitable for an X-ray structure determination, were grown from a diethyl ether solution at −25 °C. Crystals of 1 are monoclinic with space group $P2_1/n$ with four molecules in a unit cell of dimensions a = 9.7649 (4) Å, b = 9.3651 (5) Å, c = 16.0712 (6) Å, and β = 97.303 (5)°. The structure determination and refinement[7a] reveal a monomeric structure in the solid

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* To whom correspondence should be addressed.

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[6] Anal. Calcd for $C_{12}H_{19}ClN_2Sn$ (1): C, 41.72; H, 5.54; N, 8.11; Cl, 10.26; mol wt, 345.4. Found: C, 41.88; H, 5.52; N, 7.99; Cl, 10.11; mol wt, (cryoscopic in benzene), 350. Anal. Calcd for $C_{19}H_{26}N_2Sn$ (2): C, 56.89; H, 6.53; N, 6.98; mol wt, 401.1. Found: C, 56.96; H, 6.48; N, 6.81; mol wt (cryoscopic in benzene), 391. Anal. Calcd for $C_{20}H_{29}IN_2Sn$ (3): C, 44.23; H, 5.38; N, 5.16. Found: C, 44.58; H, 5.21; N, 5.03. Anal. Calcd for $C_{13}H_{22}I_2N_2Sn$ (4): C, 26.98; H, 3.83; N, 4.84. Found: C, 26.71; H, 3.74; N, 4.81.

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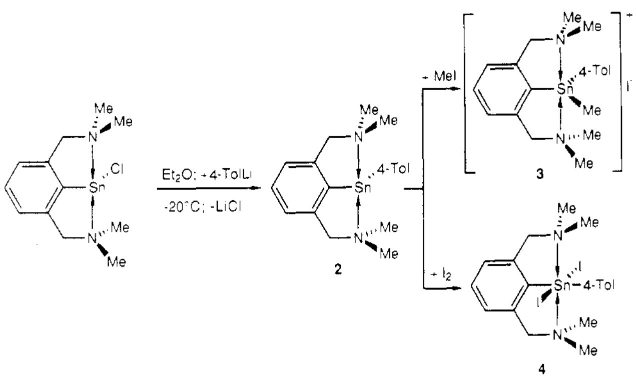
Scheme I



state (Figure 1). Relevant bond distances and angles are given in the legend to Figure 1.

In this complex the $[C_6H_3(CH_2NMe_2)_2\text{-}2,6]$ ligand is tridentate bonded to tin: σ-bonded through C(1) and bis ortho chelate bonded as a result of intramolecular Sn–N coordination of the CH_2NMe_2 substituents. The observed Sn–N distances (2.525 (8) and 2.602 (8) Å) are in the same range as reported for other divalent organotin compounds containing intramolecular coordination of neutral N donor atoms.[8a–c,11] The tin coordination sphere is completed by a Sn–Cl bond that is orientated almost orthogonal to the plane of the aryl ligand, so producing a distinctive four coordinate geometry.

The coordination stereochemistry of 1 is best described as distorted Ψ-trigonal bipyramidal with the two nitrogen atoms at the axial sites and C(1), Cl, and a tin lone pair at the equatorial sites. The reduction of the C(1)–Sn–Cl angle to 95.0 (3)°, caused by repulsive forces between the lone pair and the Sn–C and Sn–Cl bonding electrons, is fully consistent with the distorted Ψ-trigonal-bipyramidal geometry predicted by the VSEPR model[9a–c] for discrete AX_2Y_2E species (A = central atom; E = lone pair; X, Y = ligands).[10a] A similar reduction in bond angle has been found in other Ψ-trigonal-bipyramidal coordinated divalent tin compounds.[11–13]

The benzylic carbon atoms and the tin atom are in the same plane as the aryl ring, though the five-membered chelate rings are puckered with N(1) lying ca. 0.57 Å below this plane and N(2) lying ca. 0.75 Å above this plane. The N(1)–Sn–N(2) angle of 143.0 (4)° deviates considerably from the ideal value (180°) for an trigonal bipyramid, but that is not unexpected, since this value is determined by the fairly inflexible N(1)–Sn–C(1) and N(2)–Sn–C(1) bite angles of 71.9 (3)° and 71.5 (3)°, respectively.[14a]

The ^1H NMR spectrum[15] of 1 at −70 °C is consistent with a structure similar to that found in the solid. In this low-temperature spectrum there is an AB pattern for the benzylic hydrogen atoms and two resonances for the NMe_2 groups, indicating that the lone pair at the tin atom is stereochemically active. Above −65 °C separate coalescence of the NMe_2 and CH_2 resonances occurs, which may be a result of an intramolecular rearrangement process involving Sn–N dissociation/association.[17a,b] However, a process in which inversion of configuration at the Sn center occurs via a planar transition state cannot be excluded.[18a]

Interestingly, reaction of 1 with 4-tolyllithium in a 1:1 molar ratio affords after recrystallization from pentane at −78 °C {2,6-bis[(dimethylamino)methyl]phenyl}4-tolyltin(II) (2) in 80% yield (Scheme I) as a pale yellow solid. A molecular weight determination of 2 (cryoscopy in benzene) reveals that in solution it is also monomeric. On the basis of the ^1H and ^{13}C NMR spectra[15] of 2, we propose a structure which is similar to that of 1, i.e. a Ψ-trigonal-bipyramidal geometry at tin, with the bonding carbon atoms and the lone pair at the equatorial sites and both coordinating nitrogen atoms at the axial sites. To our

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(7) (a) A total of 1910 reflections ($5 < \theta < 65$; *h*, −11 to +11; *k*, 0 to 10; *l*, 0 to 18) with intensities above the $2.5\sigma(I)$ level were measured at 25 °C on a Nonius CAD4 diffractometer using graphite-monochromated Cu Kα radiation (λ = 1.5418 Å). The Sn and Cl positions were derived from a Patterson synthesis, and C and N atoms were found from a subsequent ΔF synthesis. After isotropic block-diagonal least-squares refinement, an empirical absorption correction (DIFABS)[7b] was applied (crystal dimensions 0.20 × 0.38 × 0.40 mm; μ(Cu Kα) = 158.36 cm^{-1}). Hydrogen atoms were introduced at their calculated positions. Continued refinement, anisotropic for C, Cl, N, and Sn and isotropic for H, converged to $R = 0.047$ ($R_w = 0.078$). The anomalous dispersions of Sn and Cl were taken into account. An extinction correction was applied, and a weighting scheme $w = 1/(6.46 + F_o + 0.017F_o^2)$ was employed. (b) Walker, N.; Stuart, D. *Acta Crystallogr., Sect. A: Found. Crystallogr.* **1983**, *A39*, 158.

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(10) (a) One of the reviewers has suggested that alternatively the geometry around the Sn atom can be described in terms of p and d orbitals involved in bonding and the s electron pair being nonbonding (inert s electron pair effect[10b]). This would predict a C–Sn–C angle of 90°. However, the observed scalar coupling between ^{119}Sn and $^{13}C(1)$ (350 Hz)[15] indicates the Sn–C bonds to have a considerable s character. (b) Huheey, J. E. *Inorganic Chemistry: Principles of Structure and Reactivity*; Harper & Row: New York, 1972.

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(14) (a) The N–Sn–C angles in the {2,6-bis[(dimethylamino)methyl]phenyl}methylphenyltin(IV) cation are in the same range.[16] Also in organometallic compounds in which the 2[(dimethylamino)methyl]phenyl ligand forms a five-membered chelate ring, N–M–C bite angles in the range of 70–75° are observed (see ref 14b and references cited therein). (b) van Koten, G.; Jastrzebski, J. T. B. H.; Noltes, J. G.; Pontenagel, W. M. G. F.; Kroon, J.; Spek, A. L. *J. Am. Chem. Soc.* **1978**, *100*, 5021.

(15) Relevant NMR data for **1**, **2**, **3**, and **4** (^1H and ^{13}C NMR δ relative to $SiMe_4$; ^{119}Sn δ relative to $SnMe_4$). **1**: ^1H NMR (toluene-d_8, −80 °C) δ 1.85, 2.30 (NMe_2 both s, $T_{coal.} = -65$ °C), 2.95, 3.32 (NCH_2, both d, $^2J(HH) = 12$ Hz, $T_{coal.} = -65$ °C); ^{13}C NMR (toluene-d_8, 25 °C) δ 45.5 (NMe_2), 65.8 (NCH_2), 170.7 (C(1), $^1J(^{13}C-^{119}Sn) = 350$ Hz); ^{119}Sn NMR (toluene-d_8, 25 °C) δ 155.6. **2**: ^1H NMR (toluene-d_8, −40 °C) δ 1.73, 2.11 (NMe_2 both s, $T_{coal.} = 0$ °C), 2.21 (CH_3, s), 3.23 (NCH_2, s); ^{13}C NMR (toluene-d_8, 25 °C) δ 21.7 (CH_3), 46.5 (NMe_2), 66.7 (NCH_2), 169.5 (C(1), $^1J(^{13}C-^{119}Sn) = 272$ Hz), 160.5 (C(1) tolyl, $^1J(^{13}C-^{119}Sn) = 391$ Hz); ^{119}Sn NMR (toluene-d_8, 25 °C) δ 209.6. **3**: ^1H NMR (methanol-d_4, 25 °C) δ 1.20 ($SnCH_3$, s, $^3J(^1H-^{119}Sn) = 54$ Hz), 2.18, 2.55 (NMe_2, both s), 3.90, 4.00 (NCH_2, both d, $^2J(HH) = 13$ Hz). **4**: ^1H NMR (toluene-d_8, 25 °C) δ 2.12 (CH_3, s), 2.52 (NMe_2, s), 3.68 (NCH_2, s), 8.51 (H(2) tolyl, d, $^3J(HH) = 7$ Hz, $^3J(^1H-^{119}Sn) = 120$ Hz); ^{119}Sn NMR (toluene-d_8, 25 °C) δ −208.7.

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(17) (a) Recently, the synthesis and structural characterization of $Sn(C_6H_4CH_2NMe_2\text{-}2)_2$ has been reported.[11] For this compound the coalescence of the NMe_2 resonances above −75 °C was explained in terms of fast "polytopal rearrangement". We have prepared closely related chiral $Sn[(S)\text{-}C_6H_4CH(Me)NMe_2\text{-}2]_2$[17c] for which coalescence of the diastereotopic NMe_2 resonances was observed above −45 °C in both the ^1H and ^{13}C NMR spectra. This unequivocally proves that Sn–N dissociation/association is involved in the coalescence process. (b) Inversion processes at Sn(IV) centers involving Sn–N dissociation/association have been extensively discussed by us and others, ref 17d and 17e and references cited therein. (c) Jastrzebski, J. T. B. H.; van Koten, G., manuscript in preparation. (d) Jastrzebski, J. T. B. H.; van Koten, G.; Knaap, C. T.; Schreurs, A. M. M.; Kroon, J.; Spek, A. L. *Organometallics* **1986**, *5*, 1551. (e) Jurkschat, K.; Tzchach, A.; Mügge, C.; Piret-Meunier, J.; van Meerssche, M.; van Binst, G.; Wynants, C.; Gielen, M.; Willem, R. *Organometallics* **1988**, *7*, 593.

(18) (a) A similar transition state has been proposed for the inversion of the sulfur atom in chiral spirosulfuranes.[18b] (b) Perrozi, E. F.; Martin, J. C.; Paul, I. *J. Am. Chem. Soc.* **1974**, *96*, 6735.

knowledge **2** is the first example of a mixed diaryltin(II) compound.

Compound **2** readily undergoes oxidative addition reactions. This is reflected, for example, in the instantaneous formation of {2,6-bis[(dimethylamino)methyl]phenyl}methyl(4-tolyl)tin(IV) iodide (**3**) when MeI is added to a benzene solution of **2**. Since **3** is insoluble in apolar solvents like benzene but very soluble in polar solvents like methanol, it is most likely ionic in nature. An ionic formulation for **3** (see Scheme I) is supported by its ^{1}H NMR data,[15] which are comparable with those of the earlier reported {2,6-bis[(dimethylamino)methyl]phenyl}methylphenyltin(IV) bromide, whose ionic character was unambiguously proved by an X-ray crystal structure determination.[16]

Reaction of **2** with I_2 affords, based on ^{1}H NMR data,[15] hexacoordinate *trans*-{2,6-bis[(dimethylamino)methyl]phenyl}4-tolyltin(IV) diiodide (**4**). This geometry is in accord with the observation that the two organic groups in hexacoordinated diorganotin dihalide complexes are always mutually trans.[19] In contrast a cis oxidative-addition product is obtained from the reaction of I_2 with {2,6-bis[(dimethylamino)methyl]phenyl}4-tolylplatinum(II), for which it has been proposed on steric grounds that trans oxidative-addition cannot take place.[20] Finally, it is to be noted that in the initial step of the reaction of **2** with I_2 we cannot, as yet, exclude formation of a cis oxidative-addition product, which then rearranges to the trans isomer **4**. This topic is currently under investigation.

Acknowledgment. Thanks are due to Dr. D. M. Grove for critical and stimulating discussions.

Supplementary Material Available: Tables of fractional coordinates of the non-hydrogen atoms, anisotropic thermal parameters, fractional coordinates of the hydrogen atoms, bond distances and angles of the non-hydrogen atoms, and bond distances and angles of the hydrogen atoms (7 pages); a listing of observed and calculated structure factors (9 pages). Ordering information is given on any current masthead page.

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Bonding Studies of Nitrogen Heterocyclic Ligands to (η^5-Cyclopentadienyl)ruthenium Cation: A Novel Nitrogen to π Rearrangement

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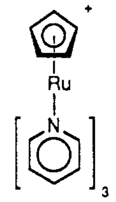
Summary: The nitrogen heterocyclic ligands, pyridine (**1**), 2-methylpyridine (**2**), 2,4-dimethylpyridine (**3**), 2,4,6-trimethylpyridine (**4**), quinoline (**5**), 2-methylquinoline (**6**), and 1,2,3,4-tetrahydroquinoline (**7**), were reacted with (η^5-cyclopentadienyl)ruthenium cation [$CpRu(CH_3CN)_3{}^+X$, X = PF_6] to determine preferences for nitrogen (N) versus π-bonding. Ligands **1**–**3** and **5** formed N-bonded complexes, while **4**, **6**, and **7** formed π- (η^6-) bonded complexes. A novel N(η^1) to π(η^6) rearrangement was discovered for the N-bonded $CpRu^+$ complexes of ligands **2**, **3**, and **5**.

The bonding mode of nitrogen heterocyclic ligands to rhodium and ruthenium complexes that have been found to act as homogeneous catalysts has been of considerable interest due to its important role in the regioselective hydrogenation of the nitrogen-containing ring of these model coal compounds.[1] Indeed, we recently reported on the bonding mode of a number of polynuclear heteroaromatic nitrogen ligands with (η^5-pentamethylcyclopentadienyl)rhodium dicationic complexes (Cp^*Rh^{2+}), i.e., nitrogen (N) versus π-bonding, and have shown that the regioselectivity of nitrogen ring reduction is in fact dependent on the ligand being N-bonded to the Rh metal center.[2]

While we were in the process of carrying out a similar bonding and catalysis study with (η^5-cyclopenetadienyl)ruthenium cation [$CpRu(S)_3{}^+$, S = CH_3CN],[3] Chaudret and Jalon published some preliminary results on the bonding mode of pyridine and several methyl-substituted pyridine ligands with (η^5-pentamethylcyclopentadienyl)ruthenium cation (Cp^*Ru^+).[4] In all cases, they isolated π-bonded complexes, while observing a pronounced solvent effect in acetone that provided a pyridine N-bonded complex [$(py)_6Ru^{2+}$], with a concomitant loss of Cp*.

We wish to report our preliminary bonding results with $CpRu(CH_3CN)_3{}^+$ and ligands **1**–**7**, which are dramatically different than the bonding study reported (pyridine and substituted analogues) for Cp^*Ru^+ and, further, we report a novel N(η^1) to π(η^6) rearrangement for N-bonded $CpRu(2\text{-methylpyridine})(CH_3CN)_2{}^+$ (**9**), $CpRu(2,4\text{-dimethylpyridine})(CH_3CN)_2{}^+$ (**10**), and $CpRu(\text{quinoline})(CH_3CN)_2{}^+$ (**14**) to their π-bonded analogues **11**, **12**, and **15**, respectively.

The reaction of excess pyridine (**1**) with $CpRu(CH_3CN)_3{}^+$ (30 min at room temperature in CH_2Cl_2) provided complex **8**, which was clearly tris N-bound from ^{1}H and ^{13}C NMR and elemental analysis data.[5] More importantly, prolonged heating (12 h) of **8** in 1,2-dichloroethane at 80 °C provided no other product (^{1}H NMR).



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The reaction of 2-methylpyridine (**2**) and 2,4-dimethylpyridine (**3**) with $CpRu(CH_3CN)_3{}^+$ also resulted in the formation of N-bonded complexes by ^{1}H NMR analysis. We were successful in isolating N-bonded complexes, **9** and **10** (R = CH_3, R_1 = H and R, R_1 = CH_3), by using short reaction times (5 min at room temperature in CH_2Cl_2), followed by addition of diethyl ether and crystallization (−30 °C) of the product.[6] Initial attempts to

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[5] Complex **8**: PF_6, 81%; ^{1}H NMR (500 MHz, $(CD_3)_2CO$, δ) Cp, 4.24 (s), H (2), 8.72 (dd, J = 6.4, 1.5 Hz), H(3), 7.46 (dd, J = 6.4, 7.5 Hz), H(4) 7.96 (tt, J = 1.5, 7.5 Hz); ratio, 5:2:2:1; $^{13}C\{^1H\}$ NMR ($(CD_3)_2CO$, δ) Cp, 70.90, C(1), 155.91, C(2), 126.52, C(3), 138.14. Anal. Calcd for $C_{20}H_{20}N_3Ru(PF_6)$: C, 43.80; H, 3.68; N, 7.66. Found: C, 43.53; H, 3.54; N, 7.40.

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