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Synthesis of aryltin(IV) complexes using arylmercury compounds. Synthesis of mixed triaryltin(IV) complexes. Crystal and molecular structure of $[\overline{Sn(2\text{-}C_6H_4N=N}Ph)Ph_2Cl]$

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Abstract

The compound $[Sn(2\text{-}C_6H_4N=NPh)Ph_2Cl]$ (**1**) has been prepared by reaction of $[SnPh_2Cl_2]$ with $[Hg(2\text{-}C_6H_4N=NPh)_2]$ (1/1) and of $[Sn(2\text{-}C_6H_4N=NPh)PhCl_2]$ with $[HgPh_2]$ (1/1). Similarly, $[Sn(AzMe)Ph_2Cl]$ (**2**) (AzMe = 5-Me-2-$(N=NC_6H_4Me\text{-}4)C_6H_3$) has been obtained by reaction of $[SnPh_2Cl_2]$ with $[Hg(AzMe)_2]$ (1/1). Metathetical reactions between **2** and KX (1/1) have been shown to give $[Sn(AzMe)Ph_2X]$ (X = Br (**3**), I (**4**), CN (**5**)]. Compound **2** does not react with NaF but the corresponding fluoro complex $[Sn(AzMe)Ph_2F]$ (**6**) can be made by treating NaF with the complex obtained from the reaction of complex **2** with $AgClO_4$. In the crystal compound **1** shows a distorted trigonal bipyramidal coordination around the tin atom. The equatorial positions are occupied by the three carbon atoms of the aryl ligands, with the tin atom lying 0.26 Å out of the equatorial plane on the side of the axial chloro atom. The 2-(phenylazo)phenyl ligand forms a five-membered chelate ring with a bite angle C–Sn–N of 70.3(1)°.

Introduction

The main advantage of organomercury compounds as transmetallating reagents over the classical organolithium and Grignard reagents is the wider range of organic

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groups, including functionalized groups that can be transferred. However, their lower reactivity restricts their utility. Thus, while the use of an excess of an aryllithium reagent allows the synthesis of polyaryltin compounds, arylmercury compounds give only mono- or di-arylspecies [1].

We have shown previously that aryltin(IV) compounds containing phenyl (Ph) and/or 2-$C_6H_4N{=}NPh$ (Ar) ligands can be made from mercury derivatives by two different routes: (i) redox transmetallation reactions [2,3]:

$$2[Hg(Ar)Cl] + Sn \rightarrow [Sn(Ar)_2Cl_2] + 2Hg$$

$$[Hg(Ar)Cl] + SnCl_2 \rightarrow [Sn(Ar)Cl_3] + Hg$$

(ii) transmetallation reactions [3,4]:

$$[Sn(Ar)Cl_3] + [HgPh_2] \rightarrow [Sn(Ar)(Ph)Cl_2] + [Hg(Ph)Cl]$$

$$[Sn(Ph)Cl_3] + [HgAr_2] \rightarrow [Sn(Ar)(Ph)Cl_2] + [Hg(Ar)Cl]$$

Triaryltin compounds, $[Sn(Ar)_3X]$, are usually obtained by (a) dearylation reactions [e.g. $[Sn(Ar)_4] + X_2$ (or XH)]; or (b) transmetallation reactions with aryl derivatives of Li, Mg, Al, or Sn [5]. We know of only one example of a mixed triaryltin(IV) complex, $[Sn(2\text{-}C_6H_4CH_2NMe_2)(Ph)_2Br]$, which was made by Van Koten by reaction of $[SnPh_2Br_2]$ with $[Cu(2\text{-}C_6H_4CH_2NMe_2)]$ [6]. In this paper we show that the organomercury route can be used to prepare mixed triaryltin(IV) compounds.

Results and discussion

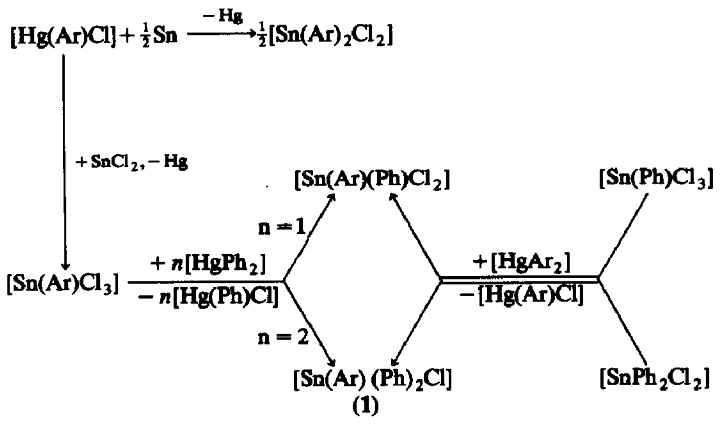
The complex $[Sn(2\text{-}C_6H_4N{=}NPh)Ph_2Cl]$ (**1**) can be obtained (i) by refluxing a chloroform solution of $[Sn(2\text{-}C_6H_4N{=}NPh)PhCl_2]$ with $[HgPh_2]$ (1/1) or (ii) by treating $[SnPh_2Cl_2]$ at room temperature in diethyl ether with $[Hg(2\text{-}C_6H_4N{=}NPh)_2]$ (1/1). Similarly, $[Sn(AzMe)Ph_2Cl]$ (**2**) (AzMe = 5-Me-2-($N{=}NC_6H_4Me$-4)C_6H_3) can be made from $[SnPh_2Cl_2]$ and $[Hg(AzMe)_2]$ (1/1).

$$[Sn(2\text{-}C_6H_4N{=}NPh)PhCl_2] + [HgPh_2] \rightarrow \underset{(\mathbf{1})}{[Sn(2\text{-}C_6H_4N{=}NPh)Ph_2Cl]} + [Hg(Ph)Cl] \qquad \text{(i)}$$

$$[SnPh_2Cl_2] + [Hg(Ar)_2] \rightarrow [Sn(Ar)Ph_2Cl] + [Hg(Ar)Cl] \qquad \text{(ii)}$$
$$(Ar = 2\text{-}C_6H_4N{=}NPh\ (\mathbf{1}),\ Ar = AzMe\ (\mathbf{2}))$$

The presence of an excess of chloride ion in transmetallation reactions is useful because this ion can bring about symmetrization of the by-products of the transmetallation reactions (e.g. $2[Hg(Ar)Cl] + Cl^- \rightarrow [HgAr_2] + [HgCl_3]^-$), so that only half as much mercurial is required [4]. Sometimes, when the aryl group is able to act as a chelating ligand (as is the case for 2-$C_6H_4N{=}NPh$ or 2-$C_6H_4CH_2NMe_2$), an anionic chloro complex is selected for the transmetallation process because then the chelating coordination of the aryl ligand leads to the formation of chloride ion and the subsequent symmetrization [7].

However, attempts to apply this approach to the synthesis of complexes **1** and **2** were unsuccessful. There was no reaction between $Me_4N[Sn(2\text{-}C_6H_4N{=}NPh)(Ph)Cl_3]$ and $[HgPh_2]$ or $Me_4N[SnPh_2Cl_3]$ and $\frac{1}{2}[Hg(AzMe)_2]$. In addition, the reaction



Scheme 1. Outline of various methods of preparing 2-(phenylazo)phenyltin(IV) complexes (Ar = 2-$C_6H_4N{=}NPh$).

$[SnPh_2Cl_2] + [Hg(2\text{-}C_6H_4N{=}NPh)Cl] + Cl^-$ gave $Me_4N[SnPh_2Cl_3]$. However, the reaction between $[Sn(Ph)Cl_3]$ and $[Hg(2\text{-}C_6H_4N{=}NPh)_2]$ (2/1) in the presence of an excess of Me_4NCl, gave $Me_4N[Sn(2\text{-}C_6H_4N{=}NPh)(Ph)Cl_3]$ [4]. The difference in the behaviour of the mono- and di-phenyltin(IV) complexes may be due to the more acidic character of the former, which could facilitate the formation of an aryl bridge between both metals in the transfer of the aryl group.

Scheme 1 summarizes the various methods we have developed for the synthesis of homo- and hetero-aryltin(IV) complexes using aryl mercury compounds [2–4].

In the reaction of complex **2** at room temperature with an excess of KX (X = Br, I, CN) the chloro ligand is replaced to give $[Sn(AzMe)Ph_2X]$ (**3**–**5**). The substitution is complete after 24 h for X = CN but not for X = Br or I. However, the reaction does not occur when complex **2** is treated with NaF (1/1, 14 h, refluxing in ethanol), and no adduct is formed with Cl^-, PPh_3, or 1,10-phenanthroline. This is

Table 1

Analytical and other data for complexes 1–6

Compound	Yield (%)	M.p. (°C)	Analytical data (found(calc.) (±0.05%)) C	H	N
$[Sn(2\text{-}C_6H_4N{=}NPh)Ph_2Cl]$ (1)	96	184	58.55 (58.90)	4.25 (3.90)	5.85 (5.70)
$[Sn(AzMe)Ph_2Cl]$ (2)	83	216	60.00 (60.35)	5.00 (4.50)	5.40 (5.40)
$[Sn(AzMe)Ph_2Br]$ (3)	87	215	56.55 (55.55)	4.50 (4.10)	4.60 (5.00)
$[Sn(AzMe)Ph_2I]$ (4)	82	202	52.00 (51.25)	4.10 (3.80)	4.10 (4.60)
$[Sn(AzMe)Ph_2CN]$ (5)	68	178	62.95 (63.80)	4.90 (4.55)	7.40 (8.25)
$[Sn(AzMe)Ph_2F]$ (6)	63	180	63.35 (62.30)	5.20 (4.65)	5.60 (5.60)

Table 2

^{1}H NMR spectroscopic data [a]

$[Sn(2\text{-}C_6H_4N{=}NPh)Ph_2Cl]$ (1)
7–8.5(m, Ph)

$[Sn(AzMe)Ph_2Cl]$ (2)
2.26 (Me), 2.51 (Me), 6.49 (d, 2H, H5, $J_{4,5}$ 8.4) 7.3 (m, 6H, Ph), 7.43 (d, 2H, H4), 7.6 (m, 5H, Ph + H2), 8.20 (d, 1H, H1, $J_{1,2}$ 1.6), 8.20 (d, 1H, H3, $J_{2,3}$ 7.8)

$[Sn(AzMe)Ph_2Br]$ (3)
2.25 (Me), 2.52 (Me), 6.97 (d, 2H, H5, $J_{4,5}$ 8.4), 7.3 (m, 6H, Ph), 7.39 (d, 2H, H4), 7.5 (m, 5H, Ph + H2), 8.20 (d, 1H, H3, $J_{2,3}$ 8), 8.23 (d, 1H, H1, $J_{1,2}$ 2)

$[Sn(AzMe)Ph_2I]$ (4)
2.24 (Me), 2.54 (Me), 6.94 (d, 2H, H5, $J_{4,5}$ 8.4), 7.3 (m, 6H, Ph), 7.32 (d, 2H, H4), 7.5 (m, 5H, Ph + H2), 8.18 (d, 1H, H3, $J_{2,3}$ 8), 8.23 (d, 1H, H1, $J_{1,2}$ 2)

$[Sn(AzMe)Ph_2CN]$ (5)
2.28 (Me), 2.52 (Me), 7.02 (d, 2H, H5, $J_{4,5}$ 8), 7.3 (m, 6H, Ph), 7.45 (d, 2H, H4), 7.5 (m, 5H, Ph + H2), 8.07 (d, 1H, H1, $J_{1,2}$ 2), 8.20 (d, 1H, H3, $J_{2,3}$ 8)

$[Sn(AzMe)Ph_2F]$ (6)
2.30 (Me), 2.47 (Me), 7.07 (d, 2H, H5, $J_{4,5}$ 8), 7.3 (m, 6H, Ph), 7.53 (m, 7H, Ph + H2 + H5), 8.02 (d, 1H, H1, $J_{1,2}$ 1), 8.16 (d, 1H, H3, $J_{2,3}$ 8)

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[a] The H atom numbering is, according to Fig. 1, C16–H1; C14–H2; C13–H3; C22–H4; C23–H5. δ (ppm), J (Hz).

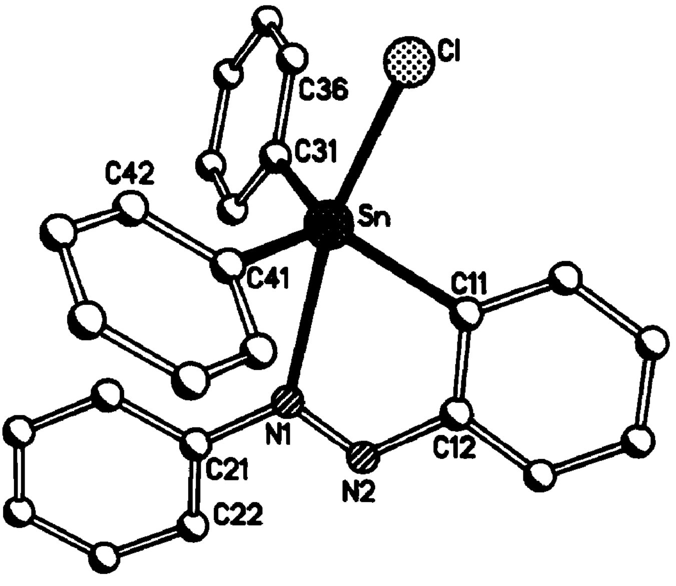
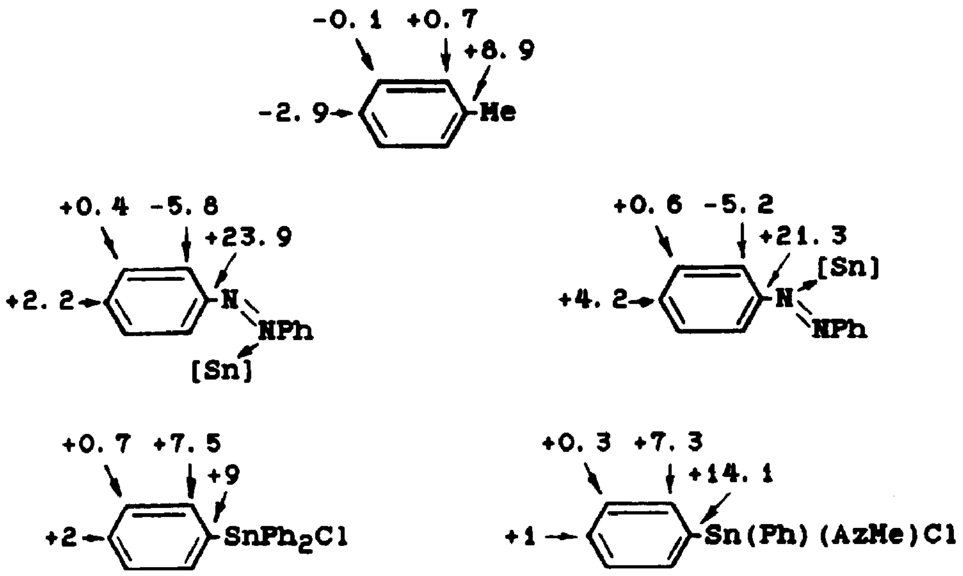


Fig. 1. The structure of complex 1 in the crystal, showing the atom numbering scheme. H atoms are omitted for clarity. Selected bond lengths (Å) and angles (°): Sn–Cl 2.445(2), Sn–N(1) 2.560(4), Sn–C(11) 2.129(4), Sn–C(31) 2.141(4), Sn–C(41) 2.130(4), N(1)–N(2) 1.253(5), N(1)–C(21) 1.437(6), N(2)–C(12) 1.424(6), Cl–Sn–N(1) 167.0(1), Cl–Sn–C(11) 97.3(1), N(1)–Sn–C(11) 70.3(1), Cl–Sn–C(31) 97.5(1), N(1)–Sn–C(31) 92.0(1), C(11)–Sn–C(31) 117.8, Cl–Sn–C(41) 96.1(1), N(1)–Sn–C(41) 85.9(1), C(11)–Sn–C(41) 113.9(1), C(31)–Sn–C(41) 123.9(1), Sn–N(1)–N(2) 113.5(3), Sn–N(1)–C(21) 131.0(3), N(2)–N(1)–C(21) 115.4(3), N(1)–N(2)–C(12) 114.5(4), N(2)–C(12)–C(11) 123.7(4), N(2)–C(12)–C(13) 114.4(4), Sn–C(11)–C(12) 118.0(3), Sn–C(11)–C(16) 124.0(3).



Scheme 2. ^{13}C NMR chemical shift parameters ($\delta(C_6H_6)$ 128.5 ppm).

typical behaviour for a triaryltin(IV) complex. Complex **2** does not react with $Na[BPh_4]$, but does react with $AgClO_4$ to give a product that analyzed as "$Sn(AzMe)(Ph)_2(ClO_4)$", but could not be isolated spectroscopically pure. However, an ethanolic solution of this complex reacted with NaF to give $[Sn(AzMe)Ph_2F]$ (**6**).

Complexes **1**–**6** are non-conducting in acetone. Table 1 presents their analytical and other data.

The mass spectra of complexes **1**–**6** show the molecular ion, except for the iodo complex **4**, and the ions $[M-X]^+$ and $[M-Ph]^+$.

Complexes **1** and **2** show the ν(Sn–Cl) band at 270(s) and 280(s) cm^{-1}, respectively. In the starting complex $[Sn(2\text{-}C_6H_4N{=}NPh)PhCl_2]$ the two bands at 340(m) and 290(s) cm^{-1} were assigned to $\nu(SnCl)_{eq}$ and $\nu(SnCl)_{ax}$, respectively [3,4]. According to this assignment the chloro ligand in complexes **1** and **2** must be in the axial position (see below). Complexes **3**–**6** do not show the ν(SnCl) band. The cyano complex **5** shows characteristic bands at 2140(w) [ν(CN)] and 330(m) (ν(CSn) or δ(SnCN)), and the fluoro complex **6** shows a ν(SnF) band at 500 cm^{-1}.

The ^1H NMR data for complexes **1**–**6** are shown in Table 2. The ^{13}C NMR chemical shifts of complexes **2**–**6** were assigned by use of the concept of additivity of substituent effects. The relevant parameters (see Scheme 2) for the substituents Me, N=N[Sn]Ph ([Sn] = $SnPh_2Cl$) and $SnPh_2Cl$ were derived from C_6H_5Me, Ph_2N_2 and $[SnPh_3Cl]$, respectively [8–10], while those for N[Sn]=NPh and Sn(Ph)(AzMe)Cl were calculated using data from complex **2**. As expected, they are not too different from the parameters for N_2Ph, and $SnPh_2Cl$, respectively. The calculated and observed values in complex **2** are almost identical. A correction of −3.3 ppm for the C(16) carbon atom (see atom numbering, Fig. 1) is necessary for complexes **3** and **4** and of −4.0 ppm for the C24 atom for the cyano complex **5**. The chemical shifts corresponding to complex **1** are also in good agreement with those calculated (±2 ppm) except that for the C11 atom, which is −4.6 ppm away from the calculated value. The chemical shifts of the Me carbon nuclei are at 21.7 and 21.3 ppm (±0.1 ppm), and that of CN at 121.6 ppm.

$^{13}C-^{117,119}Sn$ coupling is always observed for the *ortho-* (J 48 ± 1 Hz) and *meta*-carbon (J 73 ± 1 Hz) nuclei of the unsubstituted phenyl groups bonded to the tin atom. In one case (complex **6**) coupling with the *para*-carbon is observed (J ca. 20 Hz). These J values are in good agreement with those previously reported [10]. In the case of complex **6** the couplings of $^{117,119}Sn$ with those carbon atoms bonded to hydrogen of the C11–C16 ring are also observed (J(Sn, C16), ca. 30, J(Sn, C13), ca. 50, and J(Sn, C14), ca. 20 Hz).

The ^{19}F NMR spectrum of complex **6** shows a singlet at −197.1 ppm ($CFCl_3$), with $^{117,119}Sn$ satellites (J 2237 and 2140 Hz).

Crystal structure of complex 1. The structure of complex **1** is shown in Fig. 1. The tin atom is five-coordinate; the molecular geometry is that of a distorted trigonal bipyramid, with the phenyl groups equatorial and chlorine and N1 axial. The structure is related to that of the starting complex $[Sn(2\text{-}C_6H_4N{=}NPh)PhCl_2]$ [4] by replacement of the equatorial chloro ligand by a phenyl group. The disposition of the ligands is the same as in $[Sn(2\text{-}C_6H_4CH_2NMe_2)Ph_2Br]$ [11].

The chloro ligand lies almost normal (within 0.9°) to the equatorial plane defined by C11, C31 and C41, whereas the corresponding angle to N1 is 13.1° because of the limited bite of the $2\text{-}C_6H_4N{=}NPh$ ligand (C11–Sn–N1), 70.3(1)°). The tin atom lies 0.26 Å out of the equatorial plane in the direction of the Cl atom. The angles in the equatorial plane show only moderate deviations from the ideal 120° (113.9–123.9(1)°), and this is the main difference from the structure of $[Sn(2\text{-}C_6H_4N{=}NPh)PhCl_2]$ in which the C–Sn–C angle is 136.2(2)° [4]. The widening of the C–Sn–C angle was previously rationalized [4] in terms of the assumption that the hybrid orbitals involved in the Sn–C bonds would require greater *s*-character because of the more electronegative nature of the Cl atom. The replacement of this atom by a phenyl ligand in **1** is thus consistent with the narrowing of the C11–Sn–CX1 angles.

The Cl–Sn bond length (2.445(2) Å) lies in the range observed for Cl_{ax}–Sn bonds in other pentacoordinate tin complexes (2.39–2.57 Å) [12] but is longer than the 2.415(2) Å observed for Cl_{ax}–Sn in $[Sn(2\text{-}C_6H_4N{=}NPh)PhCl_2]$ [4]. This is consistent with the VSEPR * model, because the repulsion Cl_{ax}–Sn bond pair/Ph–Sn bond pair in complex **1** is greater than the corresponding Cl_{ax}–Sn bond pair/Cl_{eq}–Sn bond pair in $[Sn(2\text{-}C_6H_4N{=}NPh)PhCl_2]$.

The three C–Sn bond lengths are similar, lying in the range observed in $[Sn(2\text{-}C_6H_4CH_2NMe_2)Ph_2Br]$ [11]. The N1–Sn bond length of 2.560(4) (Å) is similar to those found in the octahedral complex $[Sn(2\text{-}C_6H_4N{=}NPh)_2Cl_2]$ (2.51, 2.58(2), [2]) or in $[Sn(2\text{-}C_6H_4N{=}NPh)PhCl_2]$ (2.541(3) Å, [4]).

The chelate ring, in which the angles are narrower than ideal values, and C13–C16 atoms are nearly coplanar (rms deviation 0.005 Å). The dihedral angle between this plane and the ring C21–C26 is 13°. The three rings C11–C16, C21–C26, and C31–C36 are perpendicular to the equatorial plane (dihedral angles 86–93°), whereas the ring C41–C46 makes a much smaller angle of 12°.

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* VSEPR = valence shell electron pair repulsion.

Experimental

Infrared spectra were recorded in the range 4000–200 cm^{-1} on a Perkin Elmer 1430 spectrophotometer with Nujol mulls between polyethylene sheets. ^1H and ^{13}C NMR spectra were recorded with $CDCl_3$ solutions (δ in ppm with respect to TMS) on a Bruker AC 200 spectrometer. The ^{19}F NMR spectrum was recorded in a Varian FT 80 (δ in ppm relative to $CFCl_3$). Conductivities were measured for ca. 10^{-4} mol dm^{-3} acetone solutions with a Philips 9501 conductimeter. Melting points were determined on a Reichert apparatus and are uncorrected. C, H and N analyses were carried out with a Perkin Elmer 240C microanalyzer. Mass spectra were determined with a Hewlett–Packard GC/MS system 5993 at 70 eV. Reactions were carried out at room temperature with magnetic stirring and without special precautions against light or atmospheric moisture unless otherwise stated.

[Sn(2-C_6H_4N=NPh)Ph_2Cl] (1). To a suspension of [$SnPh_2Cl_2$] (200 mg, 0.58 mmol) in diethyl ether (10 cm^3) was added solid [Hg(2-C_6H_4N=NPh)$_2$] (327 mg, 0.58 mmol). After 48 h the suspension was filtered to remove [Hg(2-C_6H_4N=NPh)Cl] and the resulting solution was concentrated (1 cm^3) and n-hexane (4 cm^3) added to precipitate complex **1**. Single crystals were obtained by layer diffusion of n-hexane into a diethyl ether solution of complex 1.

[Sn(AzMe)Ph_2Cl] (2). To a suspension of [$SnPh_2Cl_2$] (500 mg, 1.45 mmol) in chloroform (30 cm^3) was added [Hg(AzMe)$_2$] (900 mg, 1.45 mmol) and the mixture refluxed for 12 h. The solution was then concentrated (to 1 cm^3) and diethyl ether (50 cm^3) added to precipitate [Hg(AzMe)Cl]. The resulting suspension was filtered, the solution concentrated (to 2 cm^3), and n-hexane (10 cm^3) added to give complex **2**, which was recrystallized from diethyl ether/n-hexane.

[Sn(AzMe)Ph_2X] (X = Br (3), I (4), CN (5)). To a solution of complex **2** (100 mg, 0.19 mmol) in acetone (10 cm^3) was added solid KX (1 g, excess). After 72 h (X = Br, I) or 24 h (X = CN) the suspension was concentrated to dryness and the residue extracted with dichloromethane. The extract was filtered and the solution concentrated (1 cm^3), and n-hexane was added to precipitate complexes 3–5, which were recrystallized from diethylether/n-hexane.

[Sn(AzMe)Ph_2F] (6). To a suspension of complex **2** (100 mg, 0.19 mmol) in ethanol (15 cm^3) was added solid $AgClO_4$ (40 mg, 0.19 mmol). After 2 h in the dark the suspension was filtered through Celite, and the filtrate treated with solid NaF (24.3 mg, 0.58 mmol). After 15 h the suspension was evaporated to dryness and extracted with dichloromethane (15 cm^3). The extract was filtered then evaporated to dryness, and the solid washed with n-hexane (3 cm^3) to give **6**.

X-Ray structure determination of complex 1

Crystal data. $C_{24}H_{19}ClN_2Sn$, $M_r = 489.6$. Monoclinic, $P2_1/n$, *a* 9.3558(10), *b* 15.658(2), *c* 14.694(2) Å, β 101.540(10)°, *U* 2109 Å^3, $Z = 4$, $D_x = 1.54$ Mg m^{-3}, λ(Mo-K_α) 0.71069 Å, μ 1.35 mm^{-1}, $F(000) = 976$, *T* 293 K. Orange tablet, 0.5 × 0.4 × 0.1 mm.

Data collection and reduction. Intensities were measured in profile-fitting mode on a Stoe-Siemens four-circle diffractometer using monochromated Mo-K_α radiation ($2\theta_{max}$50°, ω/θ scans). Of 3732 measured intensities, 3705 were unique (R_{int} = 0.011) and 3017 with $F > 4\sigma(F)$ considered observed. An absorption correction based on ψ-scans was applied, with transmissions 0.72–0.89. Cell constants were refined from $\pm\omega$ values of 46 reflections in the range 2θ 20–23°.

Table 3

Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for complex 1

	x	y	z	U_{eq}
Sn	3064.6(3)	5837.8(2)	2292.4(2)	39(1)
Cl	3786(1)	5961.5(8)	787.8(7)	58(1)
N1	2764(4)	5453(2)	3937(2)	46(1)
N2	3347(4)	4757(2)	4223(3)	52(1)
C11	4073(4)	4638(3)	2686(3)	43(1)
C12	4030(4)	4322(3)	3574(3)	47(1)
C13	4675(5)	3548(3)	3892(3)	59(2)
C14	5390(5)	3080(3)	3316(4)	67(2)
C15	5429(5)	3379(3)	2443(4)	68(2)
C16	4782(5)	4161(3)	2130(3)	56(2)
C21	2077(5)	5906(3)	4584(3)	48(1)
C22	1771(5)	5524(3)	5379(3)	61(2)
C23	1091(7)	6015(4)	5954(4)	81(2)
C24	709(6)	6848(4)	5743(4)	72(2)
C25	1018(5)	7210(3)	4963(3)	67(2)
C26	1688(5)	6742(3)	4376(3)	58(2)
C31	753(4)	5855(3)	1790(3)	41(1)
C32	−251(5)	5765(3)	2361(3)	54(2)
C33	−1729(5)	5725(3)	1981(4)	69(2)
C34	−2228(5)	5777(3)	1042(4)	67(2)
C35	−1251(5)	5875(3)	482(4)	73(2)
C36	222(5)	5914(3)	846(3)	62(2)
C41	4288(4)	6907(3)	2918(3)	41(1)
C42	3842(5)	7734(3)	2704(3)	56(2)
C43	4691(7)	8412(3)	3078(4)	74(2)
C44	6003(7)	8275(4)	3656(4)	80(2)
C45	6452(6)	7461(4)	3874(3)	75(2)
C46	5607(5)	6781(3)	3513(3)	58(2)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Structure solution and refinement. The structure was solved by the heavy-atom method and refined anisotropically to $R = 0.034$, $R_w = 0.033$. Hydrogen atoms were included in the refinement using a riding model. The weighting scheme was $w^{-1} = \sigma^2(F) + 0.0002F^2$. 253 parameters, $S = 1.49$, max. Δ/σ 0.001, max. $\Delta\rho$ 1 e $Å^{-3}$. Final atomic coordinates are presented in Table 3, with selected bond lengths and angles in the caption to Fig. 1. The program system employed was XS/XLS, written by Prof. G.M. Sheldrick.

Full details of the structure determination (complete bond lengths and angles, H atom coordinates, thermal parameters, structure factors) have been deposited at the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, 7514 Eggenstein-Leopoldshafen 2, W. Germany. Any request for this material should quote a full literature citation and the reference number CSD 54407.

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References

1 See for example, R.J. Cross and J. Gemmill, J. Chem. Soc., Dalton Trans., (1984) 199; V.I. Sokolov, L.L. Troitskaya and O.A. Reutov, J. Organomet. Chem., 93 (1975) C11; R.E. Cobbledick, F.W.B. Einstein, W.R. McWhinnie and I.H. Musa, J. Chem. Res., (1979) (S) 140, (M) 1901; E. Wehman, G. van Koten, J.T.B.H. Jastrzebski, H. Ossor, and M. Pfeffer, J. Chem. Soc., Dalton Trans., (1988) 2975; J. Vicente, J. Martín, X. Soláns, and M. Font-Altaba, Organometallics, 8 (1989) 357.

2 J.L. Briansó, X. Soláns, and J. Vicente, J. Chem. Soc., Dalton Trans., (1983) 169.

3 J. Vicente, M.T. Chicote, R.M. Carreño, and M.C. Ramírez-de-Arellano, J. Organomet. Chem., 368 (1989) 263.

4 J. Vicente, M.T. Chicote, M.C. Ramírez-de-Arellano, G. Pelizzi, and F. Vitali, J. Chem. Soc., Dalton Trans., (1990) 279.

5 M. Dub (Ed.), Organometallic Compounds. Methods of Synthesis. Physical Constants and Chemical Reactions, Vol. II, R.W. Weiss (Ed.), Compounds of Germanium, Tin and Lead Including Biological Activity and Commercial Application. 2nd ed., Springer Verlag, Berlin, 1967, p. 302. First Supplement, 1973, p. 493.

6 G. van Koten, C.A. Schaap, and J.G. Noltes, J. Organomet. Chem., 99 (1975) 157.

7 J. Vicente, M.T. Chicote, M.D. Bermúdez, X. Soláns, and M. Font-Altaba, J. Chem. Soc., Dalton Trans., (1984) 557; J. Vicente, M.T. Chicote, and M.D. Bermúdez, J. Organomet. Chem., 268 (1984) 191.

8 R.M. Silverstein, G.C. Bassler, and T.C. Morrill, Spectrometric Identification of Organic Compounds, 4th ed., John Wiley, New York, 1981, p. 265.

9 H.B. Singh and W.R. McWhinnie, J. Chem. Soc., Dalton Trans., (1985) 821.

10 T.N. Mitchell, J. Organomet. Chem., 59 (1973) 189.

11 G. van Koten, J.G. Noltes, and A.L. Spek, J. Organomet. Chem., 118 (1976) 183.

12 W. Clegg, C.M.J. Grievson and K. Wade, J. Chem. Soc. Chem. Commun., (1987) 969; R.A.H. Howie, E.S. Paterson, J.L. Wardell and J.W. Burley, J. Organomet. Chem., 304 (1986) 301; J. Buckle, P.G. Harrison and J.A. Richards, J. Chem. Soc. Chem. Commun., (1972) 1104; H. Weichmann, J. Meunier-Piret and M. van Meerssche, J. Organomet. Chem., 309 (1986) 267; P.G. Harrison and K. Molloy, ibid., 152 (1978) 63; S.K. Lee and B.K. Nicholson, ibid., 309 (1986) 257; F.W.B. Einstein and B.R. Penfold, J. Chem. Soc. Chem. Commun., (1966) 780; F.W.B. Einstein and B.R. Penfold, J. Chem. Soc. A, (1968) 3019.