Synthesis of Mixed Diaryltin(IV) Complexes using an Arylmercury Compound. Crystal and Molecular Structure of $[\overline{\mathrm{Sn}(2\text{-}C_6H_4N{=}N}\mathrm{Ph})(Ph)Cl_2]$†

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The complex $[Sn(2\text{-}C_6H_4N{=}NPh)(Ph)Cl_2]$ (**1**) can be prepared by treating $[Sn(Ph)Cl_3]$ with $[Hg(2\text{-}C_6H_4N{=}NPh)_2]$ (1:1). The reactions of (**1**) with NMe_4Cl (1:1) or excess of KX salts give $NMe_4[Sn(2\text{-}C_6H_4N{=}NPh)(Ph)Cl_3]$ (**2**) or $[Sn(2\text{-}C_6H_4N{=}NPh)(Ph)X_2]$ [X = Br(**3**) or I(**4**)], respectively. The molecular structure of complex (**1**) shows a distorted trigonal bipyramidal co-ordination around the tin atom. The equatorial positions are occupied by both carbon atoms of the aryl ligands and by one of the chloro ligands. The angles in the equatorial plane differ from the 120° ideal value [C–Sn–C 136.2(2)°, Cl-Sn-C 111.4(2) and 106.0(1)°] with the tin atom lying 0.314 Å out of the equatorial plane on the side of the axial Cl atom. The 2-(phenylazo)phenyl ligand forms with the tin atom a five-membered ring with a bite angle C–Sn–N of 70.9(2)°. The Cl–Sn bond distances are normal although the axial [2.415(2) Å] is longer than the equatorial one [2.351(2) Å]. Both C–Sn bond lengths are also normal and similar [2.114(5) and 2.117(5) Å]. The N–Sn bond length [2.541(3) Å] is longer than others reported. To our knowledge, this is the first mixed diaryltin(IV) complex structurally characterized.

We have reported[1a] the synthesis and crystal structure of the complex $[Sn(2\text{-}C_6H_4N{=}NPh)_2Cl_2]$ prepared by using $[Hg(2\text{-}C_6H_4N{=}NPh)Cl]$ as transmetallating agent. The tin complex belongs to a class of organometallics, containing certain functionalized aryls, that cannot be prepared by classical transmetallation reactions due to the unavailability or instability of the corresponding organolithium or Grignard reagents. We have succeeded in preparing such complexes for gold,[2] palladium,[3] platinum,[4] and rhodium[5] by transmetallation reactions using organomercury compounds. As the use of organomercurials as intermediates in the synthesis of functionalized organometallics is still very limited[1–6] we thought it of interest to extend our study to the synthesis of other types of tin complexes.

Diaryltin compounds, $[SnR_2X_2]$, are usually obtained:[7] (*a*) by treating sodium-activated tin with RCl in the presence of ZnI_2; (*b*) by treating $(SnR_2)_n$ with X_2; (*c*) by dearylation reactions of $[SnR_4]$ or $[SnR_3X]$ with halogens; or (*d*) by arylation of SnX_4 with MgR(X) or $[SnR_3X]$ or $[SnR_4]$. In the synthesis of $[Sn(2\text{-}C_6H_4N{=}NPh)_2Cl_2]$[1a] we used a new method, $[HgR(Cl)] + Sn \longrightarrow [SnR_2X_2] + Hg$, *i.e.* a redox transmetallation reaction.

In this paper, we show that a diaryltin complex can also be obtained by arylation of a monoaryltin derivative. This reaction, although apparently obvious, requires a weak arylating agent (such as arylmercury derivatives), the classical lithium or magnesium derivatives not being the best candidates. The only precedent for this reaction is the synthesis by van Koten *et al.*[8] of $[Sn(2\text{-}C_6H_4CHZNMe_2)RX_2]$ (Z = H or Me; R = Me or Ph; X = Cl or Br) by treating $[SnRX_3]$ with $[Cu(2\text{-}C_6H_4\text{-}CHZNMe_2)]$. However, given that the synthesis of the copper reagent requires the use of the corresponding lithium derivative our method could be of wider application.

The transmetallation process we report here can be related to our arylation of monoarylgold(III) complexes with arylmercury compounds to give diarylgold(III) derivatives.[2f,h] This method allowed us to prepare mixed diarylgold(III) complexes.[2f] We also show in this paper that the 'organomercury route' is useful to prepare mixed diaryltin compounds of which we know only one example, van Koten's complex $[Sn(2\text{-}C_6H_4CH_2NMe_2)(Ph)Cl_2]$[8] the crystal structure of which is not known. To the best of our knowledge no crystal structure of such a type of complex has been reported so far.

Results and Discussion

Synthesis.—The transmetallation reaction between $[Sn(Ph)Cl_3]$ and $[Hg(2\text{-}C_6H_4N{=}NPh)_2]$ (1:1) in diethyl ether leads to the synthesis of the mixed diaryltin complex $[Sn(2\text{-}C_6H_4N{=}NPh)(Ph)Cl_2]$ (**1**) under very mild conditions (room temperature, 24 h). The mercury by-product, $[Hg(2\text{-}C_6H_4\text{-}N{=}NPh)Cl]$, separates from the reaction mixture, given its insolubility in diethyl ether. In addition, as the reaction is performed without any special precautions against oxygen or moisture the synthetic method is quite simple when compared with others using LiR, MgRX, or CuR. Complex (**1**) cannot be further arylated by $[Hg(2\text{-}C_6H_4N{=}NPh)_2]$ at room temperature.

In similar transmetallation reactions we have added an excess of Cl^- to the reaction mixture to economize on the organomercury transmetallating agent because the by-product symmetrizes $\{i.e.\ [HgR(Cl)] + \tfrac{1}{2}Cl^- \longrightarrow \tfrac{1}{2}[HgR_2] + \tfrac{1}{4}[Hg_2Cl_6]^{2-}\}$. However, if the reaction between $[Sn(Ph)Cl_3]$ and $[Hg(2\text{-}C_6H_4N{=}NPh)_2]$ is carried out at a molar ratio of 1:0.5 in the presence of excess of NMe_4Cl, instead of complex (**1**) a mixture of NMe_4Cl, $[NMe_4]_2[Hg_2Cl_6]$, and $NMe_4[Sn(2\text{-}C_6H_4N{=}NPh)(Ph)Cl_3]$ (**2**) is obtained which we were not able to separate. However, complex (**2**) can be obtained separately by treating (**1**) with NMe_4Cl (1:1, in acetone at room temperature for 6 h). Complex (**2**) behaves as a 1:1 electrolyte in acetone ($\Lambda_M = 123\ \Omega^{-1}\ cm^2\ mol^{-1}$).

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† Dichlorophenyl(2-phenylazophenyl-*CN'*)tin.

Supplementary data available: see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1990, Issue 1, pp. xix—xxii.

Table 1. Bond distances (Å) and selected bond angles (°) of complex (**1**)

Sn–Cl(1)	2.415(2)	C(5)–C(6)	1.406(11)
Sn–Cl(2)	2.351(2)	C(7)–C(8)	1.380(8)
Sn–N(1)	2.541(3)	C(7)–C(12)	1.379(6)
Sn–C(1)	2.114(5)	C(8)–C(9)	1.383(8)
Sn–C(18)	2.117(5)	C(9)–C(10)	1.378(8)
N(1)–N(2)	1.257(6)	C(10)–C(11)	1.376(11)
N(1)–C(7)	1.422(6)	C(11)–C(12)	1.379(10)
N(2)–C(13)	1.410(6)	C(13)–C(14)	1.399(8)
C(1)–C(2)	1.394(7)	C(13)–C(18)	1.402(6)
C(1)–C(6)	1.373(6)	C(14)–C(15)	1.375(9)
C(2)–C(3)	1.375(10)	C(15)–C(16)	1.364(8)
C(3)–C(4)	1.356(9)	C(16)–C(17)	1.409(9)
C(4)–C(5)	1.370(11)	C(17)–C(18)	1.381(7)

C(1)–Sn–Cl(2)	106.0(1)	Sn–C(1)–C(6)	122.3(4)
C(1)–Sn–C(18)	136.2(2)	Sn–N(1)–C(7)	132.2(3)
C(18)–Sn–Cl(2)	111.4(2)	Sn–N(1)–N(2)	112.6(3)
Cl(1)–Sn–N(1)	169.6(1)	N(2)–N(1)–C(7)	115.2(4)
Cl(1)–Sn–C(1)	100.5(1)	N(1)–C(7)–C(8)	117.5(5)
Cl(1)–Sn–C(18)	99.2(1)	N(1)–C(7)–C(12)	122.1(5)
Cl(1)–Sn–Cl(2)	93.81(8)	N(1)–N(2)–C(13)	115.2(4)
N(1)–Sn–C(1)	89.3(2)	N(2)–C(13)–C(14)	115.4(5)
N(1)–Sn–C(18)	70.9(2)	N(2)–C(13)–C(18)	123.8(4)
N(1)–Sn–Cl(2)	86.8(1)	Sn–C(18)–C(13)	117.4(3)
Sn–C(1)–C(2)	118.8(4)	Sn–C(18)–C(17)	124.0(4)

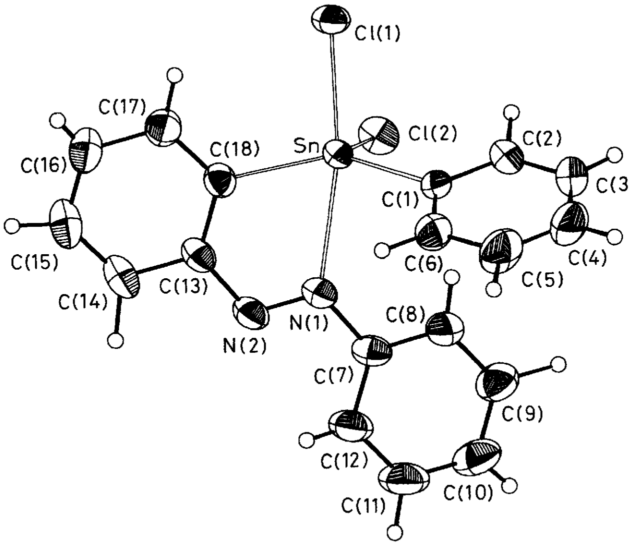

Figure. ORTEP diagram and atom labelling scheme for complex (**1**). Thermal ellipsoids are drawn at the 40% probability level

However, the reactions between complex (**1**) and excess of $\mathrm{NEt_4Br}$ (1:6 molar ratio, room temperature, 48 h) or between (**2**) and excess of KBr (1:6 molar ratio, room temperature, 5 h) do not give $[\mathrm{Sn(2\text{-}C_6H_4N{=}NPh)(Ph)Br_3}]^-$. Instead, mixtures, which are not conducting in acetone and contain (by I.R. spectroscopy) complex (**1**), the corresponding neutral bromo-complex $[\mathrm{Sn(2\text{-}C_6H_4N{=}NPh)(Ph)Br_2}]$ (**3**), and, possibly, the mixed ligand complex $[\mathrm{Sn(2\text{-}C_6H_4N{=}NPh)(Ph)ClBr}]$ were obtained. The mixture could not be separated.

Complex (**3**) and the corresponding iodo-complex $[\mathrm{Sn(2\text{-}C_6H_4N{=}NPh)(Ph)I_2}]$ (**4**) can be obtained by treating (**1**) with a large excess of KBr or KI. The reactions are carried out in acetone at room temperature for 24 h and, after removal of NaCl, the corresponding $[\mathrm{Sn(2\text{-}C_6H_4N{=}NPh)(Ph)X_2}]$ [X = Br (**3**) or I (**4**)] is precipitated with n-hexane. However, when the reaction with KBr was carried out at a 1:2 molar ratio (24 h at room temperature) a mixture of (**1**) and (**3**) was obtained.

Complex (**1**) does not react with excess of pyridine in diethyl ether at room temperature for 24 h, nor with equimolar amounts of 2,2′-bipyridine under the same conditions or even in refluxing chloroform for 2 h. Recently we have found that (**1**) can also be obtained by treating $[\mathrm{Sn(2\text{-}C_6H_4N{=}NPh)Cl_3}]$ with $\mathrm{HgPh_2}$ and that it reacts with 1,10-phenanthroline (phen) giving $[\mathrm{Sn(2\text{-}C_6H_4N{=}NPh)(Ph)Cl_2(phen)}]$.[1b]

Crystal Structure of Complex (**1**).—Complex (**1**) (see Figure) is chiral at the tin centre, both centrosymmetrically related enantiomers being present in the unit cell. Bond distances and angles are listed in Table 1. Due to the co-ordination of N(1) of the 2-(phenylazo)phenyl ligand and because there is no intermolecular association, the tin atom is five-co-ordinate. The molecular packing is dominated by van der Waals interactions. The shortest non-hydrogen contact is 3.428(7) Å between N(2) of the asymmetric unit and C(14) at $1 - x, \bar{y}, 1 - z$. The Cl(1) atom of a neighbouring molecule ($\bar{x}, \bar{y}, 1 - z$) approaches the tin atom at 3.741(2) Å. In chloroform solution the complex is also monomeric. Five co-ordination is relatively unusual for diorganotin complexes where six-co-ordination is commonly observed. Two of the few five-co-ordinated diorganotin complexes structurally characterized are $[\mathrm{SnMe_2\{BH_2(pz)_2\}Cl}]$ (pz = pyrazolyl)[9a] and $[\mathrm{SnMe_2Cl_3}]^-$.[9b,c] To the best of our knowledge,[8–10] the only five-co-ordinate diorganotin dihalide complex structurally characterized is benzothiazoledichlorodiphenyltin.[11] Some 1:1 adducts of dimethyltin dichloride (*e.g.* with salicylaldehyde,[12] phenyl(N-methyl-N-phenylamino)-sulphine,[13a] diphenylcyclopropenone,[13b] or 2,6-dimethylpyridine *N*-oxide[14]) could be viewed as[13,14] or have even been proposed to be five-co-ordinate.[12] However, short intermolecular Sn···O (3.36 Å[12,14]) or Sn···Cl bond distances [3.307(1),[13a] 3.561,[13b] and 3.399 Å[14]] suggest[14] that six-co-ordination for all these complexes is more realistic.

The molecular geometry of complex (**1**) is a distorted trigonal bipyramid. The well known rule that the most electronegative atoms (both Cl) should occupy axial positions is not fulfilled. The reason must be the bite size of the $\mathrm{2\text{-}C_6H_4N{=}NPh}$ ligand {C(18)–Sn–N(1), 70.9(2)°; average value in the complex[1a] $[\mathrm{Sn(2\text{-}C_6H_4N{=}NPh)_2Cl_2}]$, 69°} which is too small to be equatorially co-ordinated. Thus the most electronegative of the two atoms, N, which occupies the axial position, deviates from the Cl–Sn axis [Cl(1)–Sn–N(1) 169.6(1)°]. In the benzothiazole complex mentioned above,[11] where the neutral ligand is N-bonded and axial, probably for steric reasons, the Cl–Sn–N angle is 176.2°.

The equatorial sites are occupied by both carbon atoms of the aryl ligands, C(1) and C(18), and the remaining chlorine atom, Cl(2). This is the proposed solution structure for the related complex $[\mathrm{Sn(2\text{-}C_6H_4CH_2NMe_2)(Ph)Cl_2}]$[8] and the crystal structure of the benzothiazole complex.[11]

The angles in the equatorial plane are not the ideal 120° [C(1)–Sn–C(18) 136.2(2), Cl(2)–Sn–C(18) 111.4(2), and Cl(2)–Sn–C(1) 106.0(1)°] with the tin atom lying 0.314 Å out of the equatorial plane on the side of the axial Cl(1) atom. The opening of the C(1)–Sn–C(18) angle may be due to steric and electronic factors. The latter are usually rationalized assuming that the hybrid involved in the Sn–Cl bond requires an excess of *p*-orbital character, due to the greater electronegativity of the Cl atom. This would increase the *s* character of the hybrids used for the Sn–C bonds which would open the C–Sn–C angle. A similar value of this angle (132.6°) has been found in the benzothiazole complex.[11]

The Cl–Sn bond distances are normal, the axial being, as expected, longer [Cl(1)–Sn 2.415(2) Å] than the equatorial one [Cl(2)–Sn 2.351(2) Å]. The $\mathrm{Cl_{ax}}$–Sn distance in (**1**) is in the range observed (2.57—2.39 Å) for other five-co-ordinate tin complexes. Some examples in decreasing order of values are: $[\mathrm{SnMe_3\{O{=}C(Me)CH{=}PPh_3\}}]$, 2.57 Å;[15] $[\mathrm{SnMe_2Cl_3}]^-$, 2.54(3) Å;[9b,c] [Sn{$(CH_2)_3PBu^tPh$}Me_2Cl], 2.494(2) Å;[16] [$SnMe_2${$BH_2(pz)_2$}Cl], 2.488(2) Å;[9] benzothiazole complex, 2.446(1) Å,[11] [Sn{2-$C_6H_4C(Ph)$=NMe}Cl_3], 2.401(2) Å;[17] and [Sn{$CH_2CH_2C(O)OPr^i$}Cl_3], 2.389(3) Å.[18] Values of $Cl_{eq.}$–Sn bond lengths in the range 2.319(2)—2.335(1) Å have been found in five co-ordinate complexes [Sn{2-$C_6H_4C(Ph)$=NMe}Cl_3][17] and [Sn{$CH_2CH_2C(O)OPr^i$}Cl_3].[18] The axial–equatorial bond-length difference of 0.06 Å is smaller than that found (0.11 Å) in the benzothiazole complex[11] but in fairly good agreement with those reported (0.05—0.07 Å) for dichlorodimethyl(salicylaldehyde)tin,[12] [Sn{2-$C_6H_4C(Ph)$=NMe}Cl_3],[17] or [Sn{$CH_2CH_2C(O)OPr^i$}Cl_3].[18]

It is reasonable to assume, according to the valence-shell electron-pair repulsion (VSEPR) model, that $Cl_{ax.}$–Sn bond lengths in five-co-ordinate tin complexes are sensitive to the nature of the other axial and also to that of the three equatorial ligands. This leads to a range of distances wider than that of the corresponding $Cl_{eq.}$–Sn bond lengths which should depend on the nature of just the two axial ligands.

Both C–Sn bond lengths are similar [C(1)–Sn 2.114(5); C(18)–Sn 2.117(5) Å] and also normal (values of 2.112(8), 2.115(3), and 2.121(4) Å have been found in the benzothiazole complex,[11] [Sn{2-$C_6H_4C(Ph)$=NMe}Cl_3],[17] and [Sn{2-CH($SiMe_3$)$C_6H_4NMe_2$}Ph(Me)Br],[19a] respectively).

The N(1)–Sn bond length [2.541(3) Å] is similar to those found in the octahedral complex [Sn(2-C_6H_4N=NPh)$_2Cl_2$] [2.58(2) and 2.51(2) Å][1a] or in the five-co-ordinate benzothiazole complex [2.548(1) Å[11]] but longer than others reported. Thus, in the five-co-ordinate complexes [Sn{2-$C_6H_4C(Ph)$=NMe}Cl_3][17] [Sn{2-CH($SiMe_3$)$C_6H_4NMe_2$}Ph(Me)Br],[19a] or [chloro(ethyl L-cysteinato-*N*,*S*)dimethyltin][19b] the N–Sn bond lengths are 2.284(4), 2.492(3), and 2.434(12) Å, respectively.

The dihedral angle between the two co-ordinated phenyl rings is 66.0(1)°. The C(13)–C(18) ring and atoms Sn, N(1), and N(2) are nearly coplanar, the maximum displacement from the mean least-squares plane being 0.03 Å; Cl(1) is displaced out of this plane by 0.12 Å.

The hydrogen atom on C(12) is directed towards the adjacent N(2) atom, to which it seems to form an intramolecular hydrogen bond [C(12) ⋯ N(2) 2.698(7) Å; C(12)–H(12) ⋯ N(2) 97°].

Structure of Complexes (**2**)—(**4**).—The i.r. spectrum of complex (**1**) and those of (**3**) and (**4**) are almost identical in the 4 000—400 cm^{-1} region which suggests the same structure for all of them. The only differences occur in the 400—200 cm^{-1} region. The two bands appearing at 340m and 290s cm^{-1} for (**1**) should be assigned to ν(SnCl) because they do not appear for (**3**) or (**4**). According to the crystal structure of (**1**) the first band could be due to $\nu(SnCl)_{eq.}$ and the second to $\nu(SnCl)_{ax}$. A band appearing at 240m cm^{-1} for (**3**) could be assigned to ν(SnBr) because it is not present for (**1**) or (**4**).

Several attempts to prepare single crystals of complex (**2**) failed. It is reasonable to assume an octahedral geometry but the relative positions of the aryl ligands could be *cis* or *trans* according to literature data.[20]

The ^1H n.m.r. spectra of all these complexes are of very limited interest from a structural point of view. All present several complex multiplets in the range of δ 8.3—7.

Experimental

Infrared spectra were recorded in the range 4 000—200 cm^{-1} on a Perkin-Elmer 1 430 spectrophotometer using Nujol mulls between polyethylene sheets, ^1H n.m.r. spectra in $CDCl_3$ solutions (δ with respect to $SiMe_4$) on a Bruker AC 200 spectrometer. Conductivities were measured in *ca.* 10^{-4} mol dm^{-3} solutions with a Philips 9501 conductimeter. Melting points were determined on a Reichert apparatus and are uncorrected. Carbon H, and N analyses were carried out with a Perkin-Elmer 240C microanalyzer. The molecular weight of complex (**1**) was determined with a Knauer vapour-pressure osmometer in chloroform.

[Sn(2-C_6H_4N=NPh)(Ph)Cl_2] (**1**).—To a solution of [Hg(2-C_6H_4N=NPh)$_2$] (212 mg, 0.38 mmol) in diethyl ether (10 cm^3) was added liquid [Sn(Ph)Cl_3] (114 mg, 0.38 mmol). The resulting suspension was stirred for 24 h and then filtered. The filtrate was concentrated (to 1 cm^3) and n-hexane (5 cm^3) slowly added to precipitate a yellow-orange solid which was washed with n-hexane (2 cm^3) to give (**1**). (74%), m.p. 129 °C, $\Lambda_M = 0$ Ω^{-1} cm^2 mol^{-1} (Found: C, 47.85; H, 3.40; N, 6.45. $C_{18}H_{14}Cl_2N_2Sn$ requires C, 48.25; H, 3.15; N, 6.25%). *M* 463 (calc.448).

NMe_4[Sn(2-C_6H_4N=NPh)(Ph)Cl_3] (**2**).—To a solution of complex (**1**) (100 mg, 0.22 mmol) in acetone (10 cm^3) solid NMe_4Cl (24.5 mg, 0.22 mmol) was added. After 12 h the reaction mixture was filtered, the solvent evaporated (to 1 cm^3), and diethyl ether (15 cm^3) added to give complex (**2**) as a yellow solid which was recrystallized from acetone–diethyl ether (2:15, v/v). Yield: 77%. M.p. 251 °C; $\Lambda_M = 123$ Ω^{-1} cm^2 mol^{-1} (Found: C, 47.90; H, 5.00; N, 7.55. $C_{22}H_{26}Cl_3N_3Sn$ requires C, 47.40; H, 4.70; N, 7.55%).

[Sn(2-C_6H_4N=NPh)(Ph)X_2] [X = Br (**3**) *or* I (**4**).—To a solution of complex (**1**) (100 mg, 0.22 mmol) in acetone (10 cm^3) was added KX (X = Br or I) (500 mg) and the resulting suspension stirred for 24 h. The solvent was evaporated, the residue extracted with dichloromethane (10 cm^3), and filtered. The filtrate was concentrated (to 0.5 cm^3) and n-hexane (2 cm^3) added to precipitate yellow solids which were washed with n-hexane (2 cm^3) to give (**3**) or (**4**). Complex (**3**): 70%, m.p. 113 °C, $\Lambda_M = 1$ Ω^{-1} cm^2 mol^{-1} (Found: C, 40.20; H, 2.70; N, 5.45. $C_{18}H_{14}Br_2N_2Sn$ requires C, 40.25; H, 2.65; N, 5.20%). Complex (**4**): 64%, m.p. 115 °C, $\Lambda_M = 14$ Ω^{-1} cm^2 mol^{-1} (Found: C, 34.80; H, 2.30; N, 4.75. $C_{18}H_{14}I_2N_2Sn$ requires C, 34.25; H, 2.25; N, 4.45%).

Crystal Structure Determination of Complex (**1**).—A suitable crystal [obtained by liquid diffusion of n-hexane into a diethyl ether solution of (**1**)] of approximate dimensions 0.28 × 0.64 × 0.85 mm was selected and mounted on a Siemens AED single-crystal computer-controlled diffractometer with niobium-filtered Mo-K_α radiation.

A reciprocal-lattice search revealed no systematic extinctions or symmetry, indicating a triclinic lattice, the possible space groups being *P*1 and $P\bar{1}$. That the space group was centric $P\bar{1}$ was suggested by statistical tests for the normalized structure factors and later confirmed by the successful structure analysis.

Crystal data. $C_{18}H_{14}Cl_2N_2Sn$, $M = 447.92$, triclinic, $a = 11.303(3)$, $b = 8.968(3)$, $c = 9.704(3)$ Å, $\alpha = 105.66(4)$, $\beta = 83.86(2)$, $\gamma = 107.13(4)°$, $U = 904.7(5)$ A^3, space group $P\bar{1}$, $Z = 2$, $D_c = 1.644$ g cm^{-3}, $F(000) = 440$, Mo-K_α radiation, $\lambda = 0.710\,69$ Å, μ(Mo-K_α) = 17.1 cm^{-1}.

Accurate lattice and orientation parameters were determined on the basis of a least-squares analysis of the angular positions of 30 intense carefully centred reflections in diverse regions of reciprocal space. One hemisphere of data ($\pm h$, $\pm k$, $+l$) was collected at room temperature by the θ—2θ step-scan technique with a scan speed ranging from 3 to 12° min^{-1}. The 3 054 unique reflections satisfying the condition $I > 2.5\sigma(I)$ were retained from a total of 3 759 reflections measured within the limits $6.0 < 2\theta < 54.0°$. There was no evidence of crystal decomposition. The intensities were processed with the peak-profile procedure following a local modification of the Lehmann and

Table 2. Fractional atomic co-ordinates (× 10^5 for Sn, × 10^4 for Cl, N, and C) for non-hydrogen atoms

Atom	X/a	Y/b	Z/c
Sn	19 289(2)	1 704(3)	34 630(3)
Cl(1)	−61(1)	−1 811(1)	3 386(1)
Cl(2)	2 739(1)	−1 392(2)	1 445(1)
N(1)	4 123(3)	2 003(4)	3 855(4)
N(2)	4 552(3)	2 024(5)	5 005(4)
C(1)	1 459(4)	1 939(5)	2 740(4)
C(2)	1 113(5)	1 576(6)	1 324(5)
C(3)	825(6)	2 707(7)	807(7)
C(4)	877(6)	4 194(8)	1 661(8)
C(5)	1 207(7)	4 584(7)	3 059(8)
C(6)	1 512(5)	3 446(6)	3 605(6)
C(7)	4 934(4)	2 994(5)	3 029(5)
C(8)	4 602(5)	2 804(6)	1 651(5)
C(9)	5 380(6)	3 683(7)	774(7)
C(10)	6 474(6)	4 779(7)	1 294(7)
C(11)	6 783(6)	4 984(8)	2 680(8)
C(12)	6 025(5)	4 094(7)	3 557(7)
C(13)	3 762(4)	1 003(5)	5 802(5)
C(14)	4 251(5)	979(7)	7 058(5)
C(15)	3 555(6)	−2(8)	7 893(6)
C(16)	2 395(6)	−951(8)	7 502(6)
C(17)	1 891(5)	−944(7)	6 238(6)
C(18)	2 571(4)	35(5)	5 384(5)

Larsen method[21] and the data were corrected for the Lorentz and polarization effects. Neither absorption nor extinction correction was applied.

The structure was solved using a Patterson map to locate the tin atom. A Fourier difference synthesis calculated by using the phases given by tin revealed the positions of all the remaining non-hydrogen atoms. The structure was refined by full-matrix least-squares techniques to minimize the quantity $\Sigma w|\Delta F^2|$. All hydrogen atoms were located from an inner-data Fourier difference map. The final residual indices for 264 variables refined against 3 054 data were $R = 0.0416$ and $R' = 0.0511$ with a goodness of fit of 0.6594. The weighting scheme used was $w = [\sigma^2(F_o) + 0.004\,723\,F_o{}^2]^{-1}$. A final ΔF map had a peak of 1.55 e Å^{-3} near the tin atom (0.89 Å) and was featureless elsewhere.

Neutral atom scattering factors were employed and the anomalous dispersion terms for the non-hydrogen atoms were included in F_c. Computation was performed on a GOULD-SEL 32/77 computer by using the programs of the SHELX 76 package.[22] Other crystallographic computer programs used have been cited elsewhere.[23]

Final atomic co-ordinates of the non-hydrogen atoms together with their estimated standard deviations are given in Table 2.

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom co-ordinates, thermal parameters, and remaining bond distances and angles.

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