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VI*. THE PREPARATION AND MÖSSBAUER SPECTRA OF SOME BUTYLTIN(IV), DIALKYLTIN(IV) AND SIMPLE AND MIXED HEXAHALOGENOSTANNATE COMPLEXES

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SUMMARY

The Mössbauer spectra of some complexes of butyltin trichloride, dimethyltin dichloride and dioctyltin dichloride have been recorded, and the observed quadrupole splittings compared with the relative values predicted for the point-charge models. In the simple and mixed tetraethylammonium hexahalogenostannate ions, $(Et_4N^+)_2SnX_6^{2-}$ and $(Et_4N^+)_2SnX_4Y_2^{2-}$, there is a linear relationship between the isomer shift and the sum of the (Mullikan) electronegativities of the ligands.

INTRODUCTION

There has been much interest recently in correlating the structure and the Mössbauer isomer shift and quadrupole splitting parameters of organotin(IV) compounds[2-5]. These studies have been hampered by a severe lack of X-ray crystallographic data: many compounds which were once thought to be isostructurally tetrahedral are now recognised to contain 5- or 6-coordinate tin, and indeed 4-coordination appears to be the exception rather than the rule.

The most useful correlations therefore refer to trigonal bipyramidal and octahedral complexes. The magnitude of the quadrupole splitting, (ΔE), which depends upon the degree of asymmetry in the distribution of the *p*-electrons about the tin nucleus, is controlled to a large extent by the molecular asymmetry, and certain ranges of quadrupole splitting values appear to be associated with a particular configuration.

The tin atoms frequently cannot distinguish between different ligands whose donor atoms contain p_π orbitals[6], and Fitzsimmons, Seeley and Smith[2], and Parish and Platt[3] showed that, for a large variety of 6-coordinate octahedral organotin(IV) complexes of the type *trans*-R_2SnL_4 (A) and *cis*-R_2SnL_4 (B), the electric field gradients are controlled by the geometrical arrangement of the Sn–C bonds and are relatively insensitive to the nature of the group X: the quadrupole splittings are in the ratio

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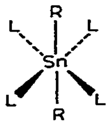
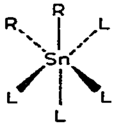
* For Part V see ref. 1.

$\Delta E_{(A)} = 2\Delta E_{(B)}$, where $\Delta E_{(A)}$ = ca. 4 mm/sec, and $\Delta E_{(B)}$ = ca. 2 mm/sec.



(A)



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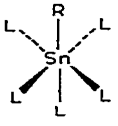

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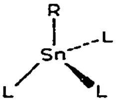

(D)

It was further proposed[2] that 6-coordinate compounds of the type $RSnL_5$ (C) should possess quadrupole splittings approximately equal to those of the compounds *cis*-R_2SnL_4 (B), *i.e.* about 2 mm/sec. As this prediction was tested by only one example $(PhSnCl_5)^{2-}(PyH)_2^+$, $\Delta E = 1.92$ mm/sec, we thought it of interest to prepare and to record the Mössbauer spectra of a number of complexes of the type $RSnL_5$, and of the type R_2SnX_4 of known structure, in order to examine further the validity of these relationships.

The isomer shift parameter (δ) can be correlated with the difference in the *s*-electron density between the source and the absorber, by the eqn. (1).

$$\delta = k \cdot \Delta |\Psi_s(0)|^2 \qquad (1)$$

In a series of tin compounds of similar structure, the isomer shift might therefore be expected to be proportional to the sum of the electronegativities of the ligands[7]. Herber investigated this relationship for a number of tin compounds which at that time were believed to be unambiguously tetrahedral[8], but many of which are now thought to have more complicated structures, while Greenwood found the correlation valid for the ions $SnCl_6^{2-}$, $SnBr_6^{2-}$, and SnI_6^{2-} (ref. 6).

The preparation and vibrational spectra of the nine possible hexahalogenostannates $SnX_4Y_2^{2-}$ (X and Y = Cl, Br, or I) have recently been reported from these laboratories[9].

$$2\ Et_4N^+Y^- + SnX_4 \rightarrow (Et_4N^+)_2\ SnX_4Y_2^{2-} \qquad (2)$$

The vibrational spectra of the simple anions (X = Y) confirm that they are regular octahedral molecules. In the mixed anions (X ≠ Y; X or Y = Cl), the groups Y are apparently in the *cis*-configuration but the bromoiodo compounds $SnBr_4I_2^{2-}$ and $SnBr_2I_4^{2-}$ decomposed in the laser beam, and the configuration of the Y_2 groups could not be specified. We have therefore taken the opportunity of determining the isomer shifts of this series of simple and mixed hexahalogenostannates, to provide a more thorough test of the electronegativity correlation.

RESULTS AND DISCUSSION

Butyltin(IV) and dialkyltin(IV) complexes

Table 1 lists the Mössbauer parameters of a series of complexes derived from butyltin trichloride [(II)–(XII)], together with the values of three complexes, Me_2SnL_4, which have recently been shown by X-ray crystallography to have a *trans*-configuration [(XIII)–(XV)] and two complexes, n-Oct_2SnL_4 [(XVI) and (XVII)] which may well have the same *trans*-structure. The values of ΔE (3.96–4.16 mm/sec) for this group of dialkyltin compounds R_2SnL_4 [(XIII)–(XVII)] are in excellent

TABLE 1

MÖSSBAUER PARAMETERS FOR ORGANOTIN COMPOUNDS

Compound[a]		Isomer shift[b] δ (mm sec)	Quadrupole splitting ΔE (mm/sec)
(I)	$BuSnCl_3$[c]	1.38	1.86
(II)	$BuSnCl_3 \cdot 2\,Ph_3PO$	1.10	2.32
(III)	$BuSnCl_3 \cdot 2$ DMA	1.10	2.22
(IV)	$BuSnCl_3 \cdot 2$ Py	0.91	1.86
(V)	$BuSnCl_3 \cdot 2$ DMSO	0.94	1.73
(VI)	$BuSnCl_3 \cdot$ Bipy	0.87	1.62
(VII)	$BuSnCl_3 \cdot$ Phen	0.91	1.60
(VIII)	$BuSnCl \cdot Ox_2$[d]	0.78	1.65
(IX)	$[BuSnCl_5]^{2-}[Et_4N]_2^+$[e]	1.12	1.86
(X)	$[BuSnCl_3Br_2]^{2-}[Et_4N]_2^+$	1.20	1.85
(XI)	$BuSnCl_3 \cdot 2$ PyO	1.02	2.00
(XII)	$BuSnOx_3$[f]	0.68	1.70
(XIII)	Me_2SnF_2[g]	1.24	4.11
(XIV)	$Me_2SnCl_2 \cdot 2$ DMSO[g]	1.40	4.16
(XV)	$Me_2SnCl_2 \cdot 2$ PyO	1.42	3.96
(XVI)	n-$Oct_2SnCl_2 \cdot$ Bipy	1.59	4.00
(XVII)	n-$Oct_2SnCl_2 \cdot$ Phen	1.56	4.11

[a] Abbreviations: DMA = *N,N*-dimethylacetamide, Py = pyridine, DMSO = dimethylsulphoxide, Phen = 1,10-phenanthroline, Ox = 8-oxyquinoline, PyO = pyridine-*N*-oxide, Bipy = 2,2'-bipyridyl and n-Oct = n-octyl. [b] Relative to SnO_2 at 77°K. [c] Ref. 5 quotes δ 1.31, ΔE 1.83 mm/sec. [d] Ref. 4 quotes δ 0.84, ΔE 1.67 mm/sec. [e] Ref. 5 quotes δ 1.07, ΔE 1.86 mm/sec. [f] Ref. 4 quotes δ 0.69, ΔE 1.82 mm/sec. [g] The presence of *trans*-methyl groups has been shown by X-ray crystallography for: (XIII)[10], (XIV)[11] and (XV)[12].

agreement with the prediction[2,3] of a quadrupole splitting of ca. 4 mm/sec for *trans* compounds of structure (A), in correlation with standard compounds.

Most of the complexes $BuSnCl_3 \cdot L_2$ [(II)–(XI)] are new compounds. All have lower isomer shifts (0.87–1.20 mm/sec) than $BuSnCl_3$ itself (1.38 mm/sec); this decrease is commonly observed in going from sp^3 to sp^3d or sp^3d^2 hybridized tin, and is taken to reflect the reduction of the *s*-electron density at the nucleus due to the shielding by the occupied 5*d* orbitals [*e.g.* representative δ values (mm/sec) are $SnCl_4$ +0.80, $SnCl_4 \cdot$Bipy +0.42; Me_2SnCl_2 +1.54, $Me_2SnCl_2 \cdot 2$DMSO +1.40]. In the series of the ionic 6-coordinate complexes $SnX_4Y_2^{2-}$ (X, Y = halide), there appears to be a linear relationship between the isomer shift and the sum of the (Mullikan) electronegativities of the six ligands (see below). The isomer shift of $BuSnCl_5^{2-}$ (1.12 mm/sec) is less than that of $BuSnCl_3Br_2^{2-}$ (1.20 mm/sec) (*cf.* $SnCl_6^{2-}$ and $SnCl_4Br_2^{2-}$, 0.50 and 0.62 mm/sec respectively), but the electronegativity relationship could not be tested further because of the difficulty in preparing other mixed halogeno complexes (see Experimental).

The quadrupole splittings for the 6-coordinate complexes [(II)–(XI)] fall in the range 1.6–2.3 mm/sec. This appears to be wider than the range of values for the *trans*- or for the *cis*-R_2SnL_4 complexes[2,3] and may result from deviations from a regular octahedral structure, or from bonding effects within the ligands which, by affecting the σ- and/or π-bonding of the ligand to the metal, thereby render different ligands electrically non-equivalent.

Parish and Platt[3] used the point-charge model to calculate that the tetrahedral 4-coordinate compounds $RSnCl_3$ (D) should have similar values of the quadrupole splitting to those of the *cis*-R_2SnL_4 complexes (B), *i.e.* ca. 2 mm/sec. The value of ΔE of 1.86 mm/sec for butyltin trichloride (I) is indeed approximately of this magnitude, as are the values for other alkyltin trihalides ($MeSnBr_3$ 1.91, $EtSnCl_3$ 1.97, $EtSnBr_3$ 1.85, $PhSnCl_3$ 1.80, $PhSnBr_3$ 1.62 mm/sec). This consistency, such as it is, may be fortuitous: no X-ray structure determination appears to have been carried out on any alkyltin trichloride, and as trimethyltin chloride and dimethyltin dichloride have been shown to be highly distorted from tetrahedral[13], some distortion would also appear to be likely in the trihalides.

The compound $BuSnOx_3$ [(XII); δ 0.68, ΔE 1.70 mm/sec] might be 7-coordinate with three bidentate Ox^- ligands, or 6-coordinate with two bidentate and one monodentate Ox^- ligand. The isomer shift is lower than that of $BuSnClOx_2$ [(VIII), δ 0.78 mm/sec] which would be compatible with the extra nuclear shielding resulting from sp^3d^3 hybridization, but might merely represent the effect of replacing Cl by a monodentate Ox^- ligand.

In all the complexes formed by ligands containing an X=O or X–O bond, the bond vibration frequency was lower in the combined than in the free ligand. The values are given in Table 2.

TABLE 2

STRETCHING FREQUENCIES OF X–O BONDS IN FREE AND COMBINED LIGANDS

Compounds	ν_{max}(X–O) (cm^{-1})		
	Free ligand	Combined ligand	Difference
$BuSnCl_3 \cdot 2\ Ph_3PO$	1190	1165	25
$BuSnCl_3 \cdot 2\ Me_2NCOMe$	1650	1600	50
$BuSnCl_3 \cdot 2\ Me_2SO$	1040	942	98
$BuSnCl_3 \cdot 2\ C_5H_5NO$	1243	1205	38

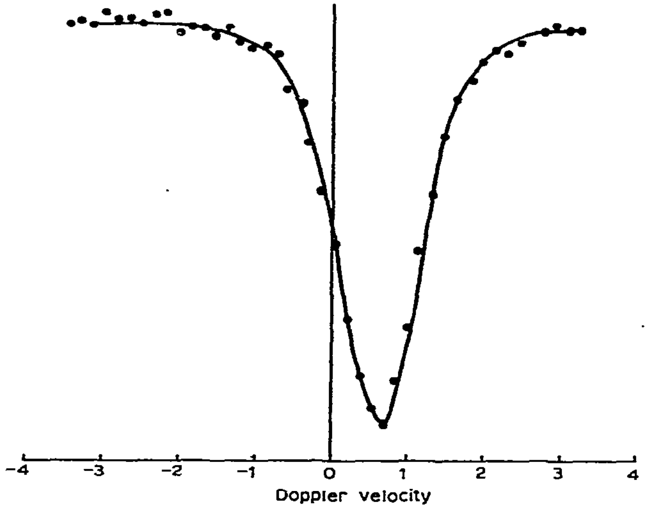

Fig. 1. Mössbauer spectrum of $[Et_4N]_2^+[SnCl_4I_2]^{2-}$.

Simple and mixed hexahalogenostannates

The third column of Table 3 lists our values for the isomer shifts of the simple and mixed tetraethylammonium hexahalogenostannates.

All the compounds gave a single peak (*e.g.* Fig. 1) of a width not greater than the natural line-width of the source. This confirms, within the limitations of the rather small differences in isomer shift which are involved, that the mixed ions $SnX_4Y_2^{2-}$ are indeed single species, and not mixtures of the two simple ions SnX_6^{2-} and SnY_6^{2-}. None showed any quadrupole coupling, implying that the various combinations of ligands do not induce enough *p*-electron asymmetry in the sp^3d^2 hybridised tin to give any significant electric field gradient. A similar absence of quadrupole coupling has been observed before in many other 6-coordinate tin compounds[6].

In Fig. 2, the isomer shifts for the various species are plotted against the sum of the (Mullikan) electronegativities of the halide ligands, and the linear relation is seen to hold within experimental error between the extremes of $SnCl_6^{2-}$ and SnI_6^{2-}. The point corresponding to the appropriate values of the SnF_6^{2-} ion lies considerably above the extrapolation of this straight line* and it would clearly be interesting to investigate the mixed fluorohalogenostannate ions $SnF_4Y_2^{2-}$ and $SnX_4F_2^{2-}$ (Y = Cl, Br, I). Dean and Evans[21] have identified ions of this type in solution by ^{19}F NMR spectroscopy, but they have apparently never been isolated in the solid state. So far, however, all our attempts to prepare complexes of the mixed fluorohalogenostannate complexes by the reaction of eqn. (2) have given only an immediate precipitation of the simple species, $(Et_4N)_2SnX_6$, in good yield, and we have not been able to isolate the mixed complexes.

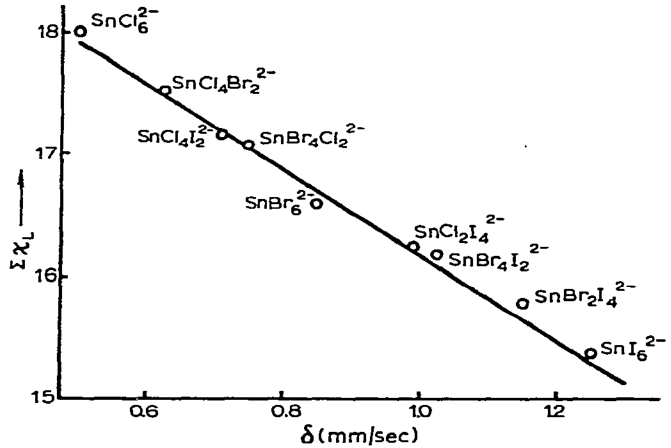

Fig. 2. Relation between the Mössbauer isomer shifts and the sum of the electronegativities of the ligands in the hexahalogenostannate ions.

Two other reports of the Mössbauer spectra of the mixed hexahalogenostannate ions have recently been published. The abstract of a lecture by Clausen and Good[22] confirms the linear relationship between isomer shifts of tetraethylammonium

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* *Note added in proof.* It has become to our attention that an X-ray study of Na_2SnF has shown that the hexafluorostannate ion is distorted from octahedral[29], this distortion may account for the anomalous value of δ.

TABLE 3

MÖSSBAUER ISOMER SHIFTS AND LIGAND ELECTRONEGATIVITIES FOR HEXAHALOGENOSTANNATE IONS, $SnX_4Y_2^{2-}$

$SnX_4Y_2^{2-}$	$\Sigma\chi_L$ (Mullikan)	δ (mm/sec) for various salts				
		Et_4N^+	Me_4N^{+a}	K^+	NH_4^+	Others
$SnCl_6^{2-}$	18.00	+0.50	+0.48	+0.50[b]		[c]
$SnCl_4Br_2^{2-}$	17.52	+0.62	+0.66			
$SnCl_4I_2^{2-}$	17.12	+0.70	+0.53		+0.54[d]	
$SnBr_4Cl_2^{2-}$	17.04	+0.74	+0.74		+0.65[d]	
$SnBr_6^{2-}$	16.56	+0.84[e]	+0.89[f]	+0.90[b]	+0.87[g]	[h]
$SnCl_2I_4^{2-}$	16.24	+0.98	+1.17			
$SnBr_4I_2^{2-}$	16.16	+1.01	+0.89			
$SnBr_2I_4^{2-}$	15.76	+1.13	+1.35			
SnI_6^{2-}	15.36	+1.25	+1.43[i]	+1.6[b]		
SnF_6^{2-}	23.56			−0.40		[j]

[a] Ref. 14. [b] Ref. 15. [c] $(MeNH_3)_2SnCl_6$ +0.50 (refs. 6 and 17). [d] Ref. 16. [e] +0.86 (ref. 17). [f] +0.84 (ref. 18). [g] Ref. 6. [h] $(Tropylium)_2SnBr_6$ +0.86; $[(p\text{-}MeC_6H_4)_2PhC]_2SnBr_6$ +0.85; $[(p\text{-}MeC_6H_4)_3C]SnBr_6$ +0.84 (ref. 18). [i] +1.25 (ref. 6). [j] Li_2SnF_6 −0.45, K_2SnF_6 −0.45, $RbSnF_6$ −0.31, $CuSnF_6$ −0.35, $SrSnF_6$ −0.32, $BeSnF_6$ −0.26, (ref. 19); K_2SnF_6 −0.50 (ref. 16); Cs_2SnF_6 −0.44 (ref. 16), −0.4 (ref. 20).

hexahalogenostannates and Mullikan electronegativities, and these authors apparently were successful in isolating also the fluoro complexes $SnX_4F_2^{2-}$ (X=Cl, Br, I) although the abstract does not list the isomer shifts.

Herber and Hwa-Shang Cheng[23] have measured the spectra of the tetramethylammonium hexahalogenostannates; their values for the isomer shifts, together with other values which are recorded in the literature, are included in Table 3. Herber and Cheng found a linear relationship between isomer shift and the sum of the Pauling electronegativities of the ligands, except for the ions $SnBr_4I_2^{2-}$ and $SnCl_4I_2^{2-}$ which they suggest may be distorted from octahedral symmetry by the large *cis*-iodo ligands; our results, and Clausen and Good's correlate better with Mullikan's values.

Consideration of the collected results in Table 3, however, suggests that any correlations of this type should be approached with caution. Herber and Cheng's values for the isomer shifts of all the tetramethylammonium iodo complexes $SnX_4Y_2^{2-}$ (X and/or Y = I) are between 0.12 and 0.22 mm/sec different from ours for the tetraethylammonium salts, which are often again different from the values for salts containing other cations. It appears that there may be a significant effect by the cation on the chemical shift[18,19], and that this must be taken into account in attempting any correlation.

EXPERIMENTAL

Mössbauer spectra were recorded with both source (SnO_2, 2 mCi) and absorber at 77°K, using a constant velocity cam-driven spectrometer, which was calibrated periodically against a β-Sn standard. A minimum of three spectra were recorded for each sample giving results which were consistent within the experimental error. Average values of δ and ΔE are quoted in Tables 1 and 3.

IR spectra were determined on Nujol mulls over the range 4000–650 cm^{-1}, using a Perkin–Elmer SP 257 instrument.

Butyltin trichloride was supplied by Albright and Wilson (Mfg) Ltd., and was distilled, b.p. 97.5–98.5°/11 mm.

The following complexes were prepared by published methods. $BuSnCl_3\cdot$Bipy (VI), m.p. 226° (lit.[24] 227–228°); $BuSnCl_3\cdot$Phen (VII), m.p. 223–225° (lit.[25] 237–239°); $BuSnClOx_2$ (VIII), m.p. 185° (lit.[26] 182–183°); $BuSnOx_3$ (XII), m.p. 229–230° (lit.[27] 229–231°). New compounds were prepared as described below.

Butyltin trichloride bis(triphenylphosphine oxide) (II)

Butyltin trichloride (1.41 g) was added slowly to a solution of triphenylphosphine oxide hemihydrate (2.87 g) in hot ethanol. The adduct separated as white crystals when the solution was cooled, and was washed thoroughly with ethanol and dried; m.p. 128°. (Found: C, 56.8; H, 5.0; Cl, 12.4. $C_{40}H_{39}Cl_3O_2P_2Sn$ calcd.: C, 57.3; H, 4.7; Cl, 12.7%.)

Butyltin trichloride bis(N,N-dimethylacetamide) (III)

Dimethylacetamide (3.48 g) and butyltin trichloride (5.64 g) were mixed warm, and allowed to stand overnight, yielding a solid crystalline mass, which could not be recrystallised. The complex was therefore washed with benzene and dried under reduced pressure, yielding white crystals, m.p. 81–82° (91% yield). (Found: C, 31.0; H, 6.0; Cl, 23.1; N, 6.4. $C_{12}H_{27}Cl_3N_2O_2Sn$ calcd.: C, 31.6; H, 6.0; Cl, 23.3; N, 6.1%.)

Butyltin trichloride dipyridine (IV)

Dry pyridine was added to a solution of butyltin trichloride until no further reaction was apparent; an instantaneous reaction occurred, yielding the complex as a white solid which was separated, washed with ether, and dried; m.p. 148–150° (96% yield). (Found: C, 37.8; H, 4.4; Cl, 23.8; N, 5.9. $C_{14}H_{19}Cl_3N_2Sn$ calcd.: C, 38.2; H, 4.4; Cl, 24.2; N, 6.4%.) Dunn and Norris[28] report m.p. 119°.

Butyltin trichloride bis(dimethylsulphoxide) (V)

An excess of dimethylsulphoxide was added to a solution of butyltin trichloride in hot ethanol. Heat was evolved, and when the solution cooled, the complex separated as white crystals, which were recrystallised from ethanol, and then from a mixture of pentane and benzene; m.p. 78–80°. (Found: C, 22.1; H, 5.0; Cl, 24.1; S, 14.6. $C_8H_{21}Cl_3O_2S_2Sn$ calcd.: C, 21.9; H, 4.8; Cl, 24.3; S, 14.6%.)

Bis(tetraethylammonium) butylpentachlorostannate (IX)

Butyltin trichloride (1.41 g) was added to tetraethylammonium chloride (1.83 g) in hot ethanol. When the solution was cool, white crystals separated, and were recrystallised from ethanol yielding the stannate in 95% yield, m.p. 193–194°. (Found: C, 38.1; H, 7.6; Cl, 28.5; N, 4.3. $C_{20}H_{49}Cl_5N_2Sn$ calcd.: C, 39.1; H, 8.0; Cl, 28.9; N, 4.6%.)

Bis(tetraethylammonium) butyldibromotrichlorostannate (X)

Tetraethylammonium bromide (4.20 g) in hot ethanol was treated with butyltin trichloride (2.82 g). On cooling, the solution deposited white crystals, which were recrystallised from ethanol yielding the stannate (65% yield), m.p. 150–151°. (Found: C, 34.2; H, 7.0; N, 4.0. $C_{20}H_{49}Br_2Cl_3N_2Sn$ calcd.: C, 34.0; H, 7.1; N, 3.9%.)

The corresponding trichlorodiiodostannate, $(Et_4N)_2BuSnCl_3I_2$ could not be obtained pure: when it was isolated, or allowed to stand in solution, it decomposed with the liberation of iodine.

Butyltin trichloride bis(pyridine N-oxide) (XI)

Butyltin trichloride (3.27 g) reacted exothermically with pyridine *N*-oxide (2.2 g) in methanol, to give a clear yellow solution. The solvent was removed under reduced pressure to give a yellow glass, which was powdered and thoroughly washed with pentane and dried, yielding the complex as a white crystalline solid, m.p. 50° (97% yield). (Found: C, 35.6; H, 4.1; Cl, 22.5; N, 5.9. $C_{14}H_{19}Cl_3N_2O_2Sn$ calcd.: C, 36.0; H, 4.1; Cl, 22.3; N, 6.0%.)

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