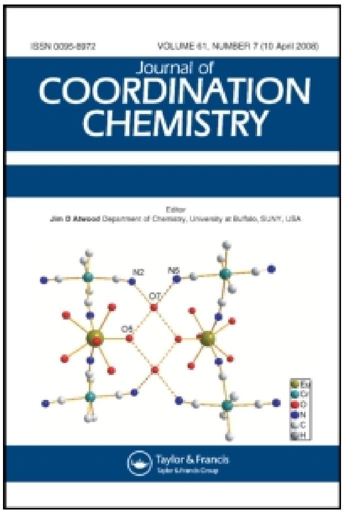

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TRICYCLOHEXYLTIN(IV) COMPLEXES WITH PICOLINIC, NICOTINIC AND 5-BROMONICOTINIC ACIDS

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(Received 17 August 1998)

A series of complexes derived from the reactions of tricyclohexyltin(IV) chloride, $(C_6H_{11})_3SnCl$ with various carboxylic acids have been synthesized and characterized respectively by using microanalytical, IR, ^1H, ^{13}C and ^{119}Sn NMR spectroscopic methods. $(C_6H_{11})_3SnCl$ reacts with picolinic, nicotinic and 5-bromonicotinic acids leading to the formation of complexes with general formula $(C_6H_{11})_3Sn(H_2O) \cdot L$ and $(C_6H_{11})_3SnCl \cdot L$ (where L = deprotonated carboxylic acid) wherein the carboxylic acid coordinates to the Sn atom *via* one of the O atoms from the COOH group.

Keywords: Tin(IV); heterocycles; acids; X-ray structure

INTRODUCTION

The synthesis of organotin(IV)–carboxylate complexes has received considerable attention during the last three decades.[1–20] Increasing interest in generating these complexes lies in the possibility to enhance biological activities associated with the respective organotin(IV) and unreacted carboxylate compounds. The hitherto known complexes have been postulated to exhibit ionic characteristics,[1,2,6–10] wherein the unidentate carboxylate moiety coordinated to the Sn atom *via* the deprotonated O atom from the OH group.

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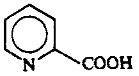
[*] Corresponding author.

In recent years, the isolation and characterization of polymeric organotin(IV)–carboxylate complexes have also been documented wherein the carboxylate moieties behave as bidentate ligands bridging between two tin moieties.[3,4,13,14]

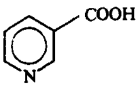
In this paper, we report the synthesis and characterization of tricyclohexyltin(IV)–carboxylate complexes formed from the reactions of $(C_6H_{11})_3SnCl$ with picolinic, nicotinic and 5-bromonicotinic acids.

The molecular structures of the carboxylic acids with their abbreviations are shown below.

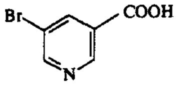
Picolinic Acid (PCOOH)



Nicotinic Acid (NCOOH)



5-Bromonicotinic Acid (5-BrNCOOH)



All the carboxylic acids were reacted with tricyclohexyltin(IV) chloride at ambient temperature. The complexes thus obtained were characterized by microanalysis, IR, ^1H, ^{13}C, ^{119}Sn NMR spectroscopic methods and a single-crystal structure analysis.

EXPERIMENTAL

Reagents

Tricyclohexyltin(IV) chloride and nicotinic acid was obtained from Aldrich Chemical Company; 2-picolinic and 5-bromonicotinic acid were obtained from Fluka Chemie AG.

Preparation of Complexes

All the complex were similarly prepared by reaction of tricyclohexyltin(IV) chloride with the appropriate carboxylic acid. The preparation of tricyclohexylaqua(nicotinato)tin(IV) is described as an example.

Tricyclohexyltin(IV) chloride (1.21 g, 3 mmol) was added to nicotinic acid (0.74 g, 6 mmol) in 95% ethanol. The mixture was refluxed for 3–4 h in inert atmosphere (under N_2 gas) followed by separation of triammonium chloride from the mixture *via* filtration. The filtrate was subsequently dried under vacuum and the compound thus isolated was recrystallized from ethanol.

Characterization

Carbon, hydrogen and nitrogen analyses were carried out on a Control Equipment Corporation model 240 XA instrument at the School of Chemical Sciences, Universiti Sains Malaysia, Penang, Malaysia. Tin was estimated using an Instrumentation Laboratory 357 atomic absorption spectrophotometer. Results are shown in Table I.

IR Spectra of the ligands and complexes (Table II) were recorded using a Perkin Elmer System 2000 FTIR spectrophotometer in the frequency range 4000–250 cm^{-1} with samples as KBr discs. ^{1}H, ^{13}C and ^{119}Sn NMR data were collected using a Bruker AC-P 300 MHz NMR spectrometer with deuterated $CDCl_3$ as solvent. Tetramethylsilane was used as internal standard in ^{1}H and ^{13}C NMR and tetramethyltin(IV) was used for ^{119}Sn NMR. Selected ^{1}H, ^{13}C and ^{119}Sn NMR peaks are presented in Tables III–V, respectively.

TABLE I Microanalytical data for PCOOH, NCOOH, 5-BrNCOOH and their complexes

Compound	*Formula*	*Calculated* (%)				*Found* (%)			
		C	*H*	*N*	*Sn*	*C*	*H*	*N*	*Sn*
$(C_6H_{11})_3SnCl\cdot[PCOOH]$	$C_{24}H_{38}NO_2ClSn$	54.7	7.3	2.7	22.9	54.9	7.6	2.6	22.5
$(C_6H_{11})_3Sn(H_2O)\cdot[NCOO]$	$C_{24}H_{39}NO_3Sn$	56.7	7.7	2.8	23.4	56.7	7.7	2.9	24.3
$(C_6H_{11})_3Sn(H_2O)\cdot[5\text{-}BrNCOO]$	$C_{24}H_{38}NO_3Sn$	49.1	6.5	2.4	20.2	49.5	6.8	2.0	21.1

TABLE II Selected IR data (cm^{-1}) for PCOOH, NCOOH, 5-BrNCOOH and their complexes

Compound	$\nu_{(COO)asym}$	$\nu_{(COO)sym}$	$\Delta\nu$
PCOOH	1717	1294	423
$(C_6H_{11})_3SnCl\cdot[PCOOH]$	1653	1341	312
NCOOH	1705	1328	377
$(C_6H_{11})_3Sn(H_2O)\cdot[NCOO]$	1613	1383	230
5-BrNCOOH	1679	1291	388
$(C_6H_{11})_3Sn(H_2O)\cdot[5\text{-}BrNCOO]$	1612	1355	257

TABLE III Selected ^{1}H chemical shift data (ppm) for $(C_6H_{11})_3SnCl$, $(C_6H_{11})_3SnCl\cdot[PCOOH]$, $(C_6H_{11})_3Sn(H_2O)\cdot[NCOO]$ and $(C_6H_{11})_3Sn(H_2O)\cdot[5\text{-}BrNCOO]$

Compound	$\delta_{(CH)aliph}$	$\delta_{(CH)arom}$
$(C_6H_{11})_3SnCl$	1.18–1.90	
$(C_6H_{11})_3SnCl\cdot[PCOOH]$	0.98–2.05	7.29–8.62
$(C_6H_{11})_3Sn(H_2O)\cdot[NCOO]$	1.24–2.30	7.29–9.22
$(C_6H_{11})_3Sn(H_2O)\cdot[5\text{-}BrNCOO]$	1.25–2.10	8.36–9.07

TABLE IV Selected ^{13}C chemical shift data (ppm) for $(C_6H_{11})_3SnCl$, $(C_6H_{11})_3SnCl \cdot [PCOOH]$, $(C_6H_{11})_3Sn(H_2O) \cdot [NCOO]$ and $(C_6H_{11})_3Sn(H_2O) \cdot [5\text{-}BrNCOO]$

Compound	$\delta_{(CH)aliph}$	$\delta_{(CH)arom}$	$\delta_{(COO)}$
$(C_6H_{11})_3SnCl$	26.7–33.7		
$(C_6H_{11})_3SnCl \cdot [PCOOH]$	23.6–35.8	125.2–136.6	150.0
$(C_6H_{11})_3Sn(H_2O) \cdot [NCOO]$	26.8–36.3	130.0–152.2	169.5
$(C_6H_{11})_3Sn(H_2O) \cdot [5\text{-}BrNCOO]$	26.8–34.3	140.1–150.0	153.4

TABLE V ^{119}Sn chemical shift data (ppm) for $(C_6H_{11})_3SnCl$, $(C_6H_{11})_3SnCl \cdot [PCOOH]$, and $(C_6H_{11})_3Sn(H_2O) \cdot [NCOO]$

Compound	δ
$(C_6H_{11})_3SnCl$	72.43
$(C_6H_{11})_3SnCl \cdot [PCOOH]$	22.15, 71.95
$(C_6H_{11})_3Sn(H_2O) \cdot [NCOO]$	25.86

Crystallography: Determination of the X-ray Crystal Structure of Tricyclohexylaqua(nicotinato)tin(IV)

Intensity data for a crystal with dimensions $0.5 \times 0.3 \times 0.2$ mm were measured at room temperature on a Siemens P4 diffractometer fitted with graphite-monochromated MoKα radiation, $\lambda = 0.71073$ Å. The θ–2θ scan technique was employed to measure a total of 23314 reflections such that $\theta_{max} < 27.5°$. There were 9620 unique data, of which 7572 satisfied the $F > 4\sigma(F)$ criterion of observability and were used in the subsequent analysis. The structure was solved by using Siemens SHELXTL Plus[21] and refined by a full-matrix least-squares procedure. All non-hydrogen atoms were refined with anisotropic thermal parameters and hydrogen atoms were included in their idealized positions (C–H = 0.96 Å) and refined isotropically. A weighing scheme of the form $w = k/(\sigma^2(F) + gF^2)$ was used and the refinement continued to final $R = 0.043$, $R_w = 0.049$ for $k = 1.000$ and $g = 0.0002$. The analysis of variance showed no special features and the maximum residual electron density peak in the final difference map was $0.77\, e\, Å^3$. The scattering factors for all the atoms were used as incorporated in SHELX76.[22] Crystal data and refinement parameters are summarized in Table VI and bond lengths and angles in Table VII. Lists of atomic coordinates, thermal parameters and observed and calculated structure factors have been deposited as supplementary material with the Cambridge Crystallographic Data Centre, and are available on request from the authors.

TABLE VI Crystal data for $(C_6H_{11})_3Sn(H_2O)\cdot[NCOO]$

Empirical formula	$C_{24}H_{39}NO_3Sn$
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	$a = 12.100(2)$ Å
	$b = 12.1352(16)$ Å
	$c = 19.090(4)$ Å
	$\alpha = 76.069(15)°$
	$\beta = 76.177(11)°$
	$\gamma = 68.242(12)°$
Volume	2491.8(8) Å^3
z	4
Formula weight	508.3
Density (calc.)	1.355 mg m^{-3}
Absorption coefficient	1.048 mm^{-1}
$F(000)$	1056

TABLE VII Bond distances (Å) and angles (°) with e.s.d.'s in parentheses for non-hydrogen atoms

SnA–O(1A)	2.172(3)	SnA–O(3A)	2.452(3)
SnA–C(1A)	2.162(4)	SnA–C(7A)	2.144(5)
SnA–C(13A)	2.158(3)	N(1A)–C(23A)	1.319(6)
N(1A)–C(25A)	1.318(6)	O(1A)–C(19A)	1.275(4)
O(2A)–C(19A)	1.234(6)	C(1A)–C(2A)	1.460(9)
C(1A)–C(6A)	1.491(6)	C(2A)–C(3A)	1.537(9)
C(3A)–C(4A)	1.446(9)	C(4A)–C(5A)	1.476(11)
C(5A)–C(6A)	1.518(9)	C(7A)–C(8A)	1.515(5)
C(7A)–C(12A)	1.478(7)	C(8A)–C(9A)	1.520(10)
C(9A)–C(10A)	1.461(10)	C(10A)–C(I1A)	1.481(8)
C(11A)–C(12A)	1.543(10)	C(13A)–C(14A)	1.540(7)
C(13A)–C(18A)	1.453(7)	C(14A)–C(15A)	1.535(7)
C(15A)–C(16A)	1.403(10)	C(16A)–C(17A)	1.542(12)
C(17A)–C(18A)	1.539(8)	C(19A)–C(20A)	1.486(6)
C(20A)–C(21A)	1.358(5)	C(20A)–C(25A)	1.386(6)
C(21A)–C(22A)	1.383(8)	C(22A)–C(23A)	1.367(9)
SnB–O(1B)	2.180(3)	SnB–O(3B)	2.463(3)
SnB–C(1B)	2.151(4)	SnB–C(11B)	2.160(3)
SnB–C(13B)	2.150(5)	N(1B)–C(23B)	1.314(6)
N(1B)–C(25B)	1.322(6)	O(1B)–C(19B)	1.274(4)
O(23)–C(19B)	1.218(6)	C(1B)–C(2B)	1.489(7)
C(1B)–C(6B)	1.478(9)	C(2B)–C(3B)	1.527(9)
C(3B)–C(4B)	1.479(11)	C(4B)–C(5B)	1.454(9)
C(5B)–C(6B)	1.532(9)	C(7B)–C(8B)	1.524(7)
C(7B)–C(11B)	1.524(7)	C(8B)–C(12B)	1.409(10)
C(9B)–C(10B)	1.559(8)	C(9B)–C(12B)	1.537(12)
C(I0B)–C(11B)	1.465(7)	C(13B)–C(14B)	1.517(6)
C(13B)–C(18B)	1.471(7)	C(14B)–C(15B)	1.536(10)
C(15B)–C(16B)	1.454(10)	C(16B)–C(17B)	1.468(8)
C(17B)–C(18B)	1.547(10)	C(19B)–C(20B)	1.504(6)
C(20B)–C(21B)	1.362(5)	C(20B)–C(25B)	1.379(6)
C(21B)–C(22B)	1.382(8)	C(22B)–C(23B)	1.365(8)

TABLE VII (*Continued*)

O(1A)-SnA-O(3A)	176.3(1)	O(1A)-SnA-C(1A)	91.5(1)
O(3A)-SnA-C(1A)	85.3(1)	O(1A)-SnA-C(7A)	98.5(1)
O(3A)-SnA-C(7A)	84.6(1)	C(1A)-SnA-C(7A)	115.5(2)
O(1A)-SnA-C(13A)	99.0(1)	O(3A)-SnA-C(13A)	80.6(1)
C(1A)-SnA-C(13A)	113.7(2)	C(7A)-SnA-C(13A)	126.8(2)
C(23A)-N(1A)-C(25A)	117.5(4)	SnA-O(1A)-C(19A)	118.8(3)
SnA-C(1A)-C(2A)	114.3(3)	SnA-C(1A)-C(6A)	114.3(4)
C(2A)-C(1A)-C(6A)	113.0(5)	C(1A)-C(2A)-C(3A)	112.8(5)
C(2A)-C(3A)-C(4A)	113.2(6)	C(3A)-C(4A)-C(5A)	113.3(6)
C(4A)-C(5A)-C(6A)	112.7(5)	C(1A)-C(6A)-C(5A)	113.3(5)
SnA-C(7A)-C(8A)	114.1(4)	SnA-C(7A)-C(12A)	114.0(3)
C(8A)-C(7A)-C(12A)	111.8(4)	C(7A)-C(8A)-C(9A)	112.7(5)
C(8A)-C(9A)-C(10A)	113.7(5)	C(9A)-C(10A)-C(11A)	113.3(5)
C(10A)-C(11A)-C(12A)	112.3(6)	C(7A)-C(12A)-C(11A)	111.9(4)
SnA-C(13A)-C(14A)	113.2(3)	SnA-C(13A)-C(18A)	117.7(3)
C(14A)-C(13A)-C(18A)	111.0(4)	C(13A)-C(14A)-C(15A)	109.8(5)
C(14A)-C(15A)-C(16A)	114.9(5)	C(15A)-C(16A)-C(17A)	111.2(5)
C(16A)-C(17A)-C(18A)	109.7(6)	C(13A)-C(18A)-C(17A)	112.5(5)
O(1A)-C(19A)-O(2A)	123.2(4)	O(1A)-C(19A)-C(20A)	116.3(4)
O(2A)-C(19A)-C(20A)	120.4(3)	C(19A)-C(20A)-C(21A)	121.1(4)
C(19A)-C(20A)-C(25A)	121.1(3)	C(21A)-C(20A)-C(25A)	117.8(4)
C(20A)-C(21A)-C(22A)	118.9(5)	C(21A)-C(22A)-C(23A)	118.6(4)
N(1A)-C(23A)-C(22A)	123.0(5)	N(1A)-C(25A)-C(20A)	123.8(3)
O(1B)-SnB-O(3B)	176.2(1)	O(13)-SnB-C(1B)	91.6(1)
O(3B)-SnB-C(1B)	85.2(1)	O(1B)-SnB-C(11B)	99.0(1)
O(3B)-SnB-C(11B)	80.5(1)	C(1B)-SnB-C(11B)	114.2(2)
O(1B)-SnB-C(13B)	98.8(1)	O(3B)-SnB-C(13B)	84.5(1)
C(1B)-SnB-C(13B)	115.2(2)	C(11B)-SnB-C(13B)	126.6(2)
C(23B)-N(1B)-C(25B)	117.8(4)	SnB-O(1B)-C(19B)	118.1(3)
SnB-C(1B)-C(2B)	113.9(4)	SnB-C(1B)-C(6B)	113.5(3)
C(2B)-C(1B)-C(6B)	113.2(5)	C(1B)-C(2B)-C(3B)	112.9(5)
C(2B)-C(3B)-C(4B)	112.2(5)	C(3B)-C(4B)-C(5B)	113.8(6)
C(4B)-C(5B)-C(6B)	113.6(6)	C(1B)-C(6B)-C(5B)	111.9(5)
C(8B)-C(7B)-C(11B)	110.9(5)	C(7B)-C(8B)-C(12B)	114.5(5)
C(10B)-C(9B)-C(12B)	109.5(6)	C(9B)-C(10B)-C(11B)	111.4(5)
SnB-C(11B)-C(7B)	113.9(3)	SnB-C(11B)-C(10B)	117.0(3)
C(7B)-C(11B)-C(10B)	111.3(4)	C(8B)-C(12B)-C(9B)	111.6(5)
SnB-C(13B)-C(14B)	113.6(4)	SnB-C(13B)-C(18B)	114.1(3)
C(14B)-C(13B)-C(18B)	112.0(4)	C(13B)-C(14B)-C(15B)	112.3(5)
C(14B)-C(15B)-C(16B)	112.8(5)	C(15B)-C(16B)-C(17B)	113.0(5)
C(16B)-C(17B)-C(18B)	112.8(6)	C(13B)-C(18B)-C(17B)	111.4(4)
O(1B)-C(19B)-O(2B)	124.4(4)	O(1B)-C(19B)-C(20B)	115.6(4)
O(2B)-C(19B)-C(20B)	120.1(3)	C(19B)-C(20B)-C(21B)	121.1(4)
C(19B)-C(20B)-C(25B)	121.2(3)	C(21B)-C(20B)-C(25B)	117.7(4)
C(20B)-C(21B)-C(22B)	119.3(5)	C(21B)-C(22B)-C(23B)	118.3(4)
N(1B)-C(23B)-C(22B)	123.3(5)	N(1B)-C(25B)-C(20B)	123.5(3)

RESULTS AND DISCUSSION

The molecular structure of tricyclohexylaqua(nicotinato)tin(IV) is shown in Figure 1. The structure shows tin to be five-coordinated, with planar

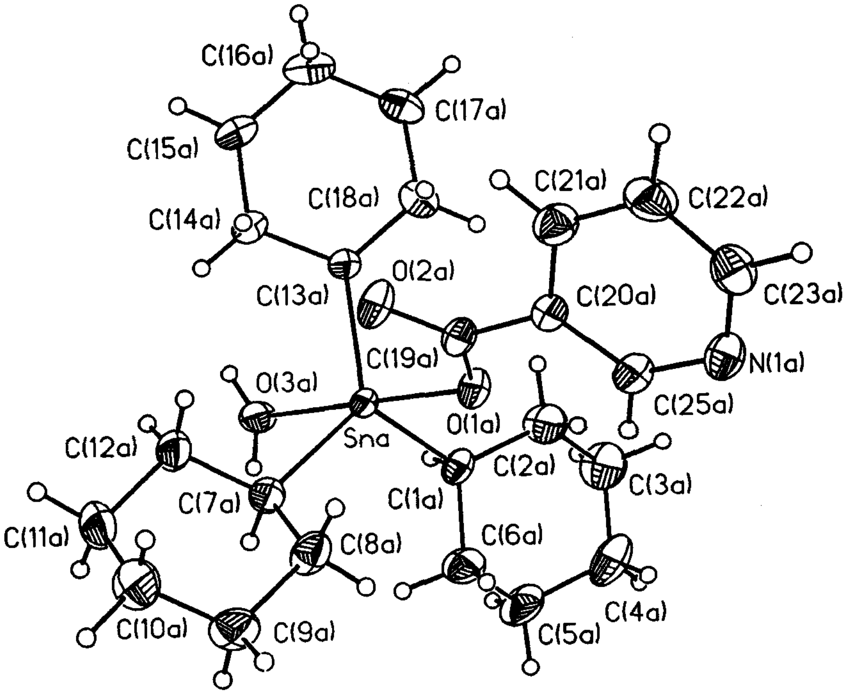


FIGURE 1 Crystal structure of $(C_6H_{11})_3Sn(H_2O)\cdot[NCOO]$ showing the atom labelling scheme.

cyclohexyl-groups, and axial positions being occupied by oxygen from a unidentate carboxylated group and a water molecule. The $Sn-O(H_2O)$ bond distance (2.452(3) Å) (Table VII) is longer than that in trimethyltin (2-pyridylcarboxylate) monohydrate (2.43(3) Å)[6] by 0.02 Å. The elongation of the $Sn-O(H_2O)$ bond can be ascribed to two factors: the stronger electron donating properties of the cyclohexyl-groups, and the bulkiness of the saturated cyclohexyl-rings in comparison with methyl groups. The C=O bond distance, 1.234 Å, compares well with similar bond distances found in $(C_6H_{11})_3SnO_2CCH_3$ (1.25 Å)[23] and $(CH_3)_3SnO_2CCH_3$ (1.24 Å).[24]

The participation of the carboxylate group in the new coordination mode can also be inferred from the IR data (Table II). A band assignable to the asymmetrical carboxylic anion COO^- is shifted markedly to lower frequency from 1705 to 1613 cm^{-1} suggesting coordination *via* the O atom from the deprotonated hydroxyl group as reported for trimethyltin (2-pyridylcarboxylate) monohydrate.[6]

From the ^{1}H NMR spectrum of tricyclohexylaqua(nicotinato)tin(IV) (Table III), it clearly shows that there are four well-resolved peaks centering at $\delta = 7.33$, 8.26, 8.67 and 9.22 ppm, which can be ascribed to four aromatic protons. The peaks assignable to the cyclohexyl protons are observable within $\delta = 2.3-1.2$ ppm. The structure of tricyclohexylaqua(nicotinato)-tin(IV) can also be supported by the ^{13}C NMR which shows the occurrence of five peaks from 122 to 154 ppm (Table IV) indicating the presence of five distinct aromatic carbons. Whilst the peaks occurring at $\delta = 76.5-77.5$ ppm can be ascribed to the cyclohexyl carbons, the carbon of the carboxylated functional group is observed at $\delta = 169.5$ ppm. The presence of the new Sn–O coordination mode in the complex can also be substantiated by the occurrence of a new peak at $\delta = 25.86$ ppm in the ^{119}Sn NMR spectrum (Table V). The disappearance of a peak at $\delta = 72.43$ ppm attributable to the Sn–Cl in cyclohexyltin(IV) chloride further indicates that the chloride ion is no longer attached to the Sn atom.

Similar observations as inferred from the spectroscopy data has also been found for tricyclohexylaqua(picolinic)tin(IV) and tricyclohexylaqua (5-bromonicotinato)tin(IV) indicating the acid ligands coordinating to the central Sn atom *via* one of the O atoms from the COOH group and the complexes thus obtained possess five-coordinated tin atoms.

However, inspection of the ^{119}Sn NMR data shows that a peak attributable to Sn–Cl at $\delta = 72.43$ ppm in the uncoordinated tricyclohexyltin(IV) chloride has also shifted upfield but only over a very small margin in comparison with that in tricyclo-hexylaqua(picolinato)tin(IV) ($\delta = 71.95$ ppm). One of the factors which can account for this observation is that the chloride ion attached to the central Sn atom remains almost intact upon complexation.

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References

[1] R. Okawa, D.E. Webster and E.G. Rochow, *J. Am. Chem. Soc.* **82**, 3287 (1960).
[2] B.F.E. Ford, B.V. Liengme and J.R. Sams, *Chem. Comm.* 1333 (1968).
[3] B.F.E. Ford, B.V. Liengme and J.R. Sams, *J. Organomet. Chem.* **19**, 53 (1969).
[4] D.W. Allen, J.S. Brooks, R. Formstone, A.J. Crowe and P.J. Smith, *J. Organomet. Chem.* **156**, 359 (1978).

[5] W.D. Honnick J.J. Zuckerman, *J. Organomet. Chem.* **178**, 133 (1979).

[6] P.G. Harrison and R.C. Philips, *J. Organomet. Chem.* **182**, 37 (1979).

[7] S. Calogero, P. Ganis, V. Peruzzo and G. Tagliavini, *J. Organomet. Chem.* **191**, 381 (1980).

[8] L. Prasad, E.J. Gabe and F.E. Smith, *Acta Cryst.* **B38**, 1325 (1982).

[9] J.F. Vollano, R.O. Day, D.N. Rau, V. Cahndrasekhar and R.R. Holmes, *Inorg. Chem.* **23**, 3153 (1984).

[10] R.G. Swisher, J.F. Vollano, V. Chandrasekhar, R.O. Day and R.R. Holmes, *Inorg. Chem.* **23**, 3147 (1984).

[11] K.C. Molloy, T.G. Purcell, H. Schumann and J.J. Zuckerman, *Organometallics* **5**, 85 (1986).

[12] J. Holecek, K. Handlir, M. Nadvornik and A. Lycka,· *J. Organomet. Chem.* **258**, 147 (1983).

[13] K.C. Molloy, K.Quill and I.W. Nowell, *J. Chem. Soc. Dalton Trans.* 101 (1987).

[14] S.W. Ng, C. Wei and V.G. Kumar Das, *J. Organomet. Chem.* **345**, 59 (1988).

[15] G.K. Sandhu, N. Sharma and E.R.T. Tiekink, *J. Organomet. Chem.* **371**, C1 (1989).

[16] G.K. Sandhu, S.P. Verma and E.R.T. Tiekink, *J. Organomet. Chem.* **393**, 195 (1990).

[17] E.R.T. Tiekink, *Appl. Organomet. Chemistry*, **5**, 1 (1991).

[18] S.W. Ng, V.G. Kumar Das and E.R.T. Tiekink, *J. Organomet. Chem.* **403**, 111 (1991).

[19] C. Vatsa, V.K. Jain, T.K. Das and E.R.T. Tiekink, *J. Organomet. Chem.* **421**, 21 (1991).

[20] S.G. Teoh, S.H. Ang, E.S. Looi, C.A. Keok, S.B. Teo and H.K. Fun, *J. Organomet. Chem.* **527**, 15 (1997).

[21] G.M. Sheldrick, SHELXTL PC^{TM} Release 4.1 (Siemens Crystallographic Research Systems, Madison, USA, 1990).

[22] G.M. Sheldrick, SHELX-76. A Program for Crystal Structure Determination (University of Gottingen, Germany, 1976).

[23] P.G. Harrison, T.J. King and M.A. Healy, *J. Organomet. Chem.* **182**, 17 (1979).

[24] N.W. Alcock and R.E. Timms, *J. Chem. Soc.* (*A*), 1873 (1968).