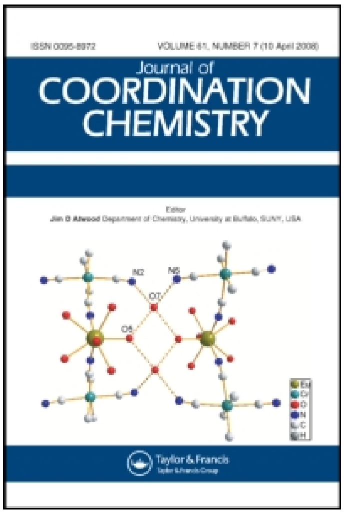

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SYNTHESIS AND CRYSTAL STRUCTURE OF POLYMERIC TRIPHENYLTIN(IV) 4-PYRIDINECARBOXYLATE

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SYNTHESIS AND CRYSTAL STRUCTURE OF POLYMERIC TRIPHENYLTIN(IV) 4-PYRIDINECARBOXYLATE

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A crystal structure determination of the title compound reveals that it has a one-dimensional polymeric structure co-crystallized with molecules of the crystallization solvent, toluene, in 1 : 1 ratio in the crystal lattice. Each of the Sn atoms exists in a distorted trigonal bipyramidal geometry with the axial positions occupied by the O atom of the carboxylate group and the N atom of the pyridyl ring of the neighbouring molecule. The axial N′–Sn–O connection makes an angle approaching linearity (175.5(1)°).

Keywords: Triphenyltin(IV) 4-pyridinecarboxylate; X-ray structure; coordination polymer

INTRODUCTION

The study of the structural chemistry of triorganotin carboxylates of the empirical formula $R_3Sn(O_2CR')$ has received considerable attention owing to the various types of structure that have been found in the solid state for these compounds.[1–5] Triorganotin carboxylates are generally polymeric because of the bidentate bridging carboxylate ligands which result in the *trans*-R_3SnO_2 trigonal bipyramidal geometry at tin atoms, especially in the absence of other donor atoms in the carboxylate unit.[6–9] However, organotin compounds with bulky R groups coordinated to tin or of the carboxylic acid tend to favour monomeric structures.[3,8–12]

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From the crystal structure determination, trimethyltin(IV) 2-pyridinecarboxylate monohydrate, $[(CH_3)_3Sn(O_2CC_5H_4N)] \cdot H_2O$ adopts the *trans*-R_3SnO_2 structural type with carboxylate-bridging.[13] In this compound, the pyridine N atom of the carboxylate ligand does not coordinate to the tin atom but connects to a water molecule of crystallization *via* a hydrogen bond. However, for triphenyltin(IV) 3-pyridinecarboxylate, the pyridine N atom functions as a donor atom, coordinating to the neighbouring tin atom and leading to a nitrogen-bridged polymeric structure.[14] Nitrogen-bridged structures have also been inferred for tri-*n*-butyltin(IV) 3- and 4-pyridinecarboxylates on infrared and Mössbauer spectroscopic evidence.[15] Here, we report the synthesis and a crystal structure study of triphenyltin 4-pyridinecarboxylate as it is of interest to determine the influence of the pyridine N atom which is now at the *para* position of the carboxylic acid.

EXPERIMENTAL

General and Instrumental

Bis(triphenyltin) oxide and 4-pyridinecarboxylic (isonicotinic) acid were purchased from the Aldrich Chemical Company. Microanalysis was carried out on a Control Equipment Corporation model 240XA elemental analyser at the School of Chemical Sciences, Universiti Sains Malaysia, Penang, Malaysia. The IR absorption spectrum was recorded using a Perkin-Elmer FTIR 1650 spectrophotometer in the frequency range 4000–450 cm^{-1} with the sample prepared in KBr disc. ^1H, ^{13}C and ^{119}Sn NMR spectra of the sample dissolved in DMSO-d_6 were recorded on a Bruker 300 MHz AC-P NMR spectrometer.

Synthesis of Triphenyltin(IV) 4-Pyridinecarboxylate

Bis(triphenyltin) oxide (1.00 g, 1.4 mmol), 4-pyridinecarboxylic acid (0.34 g, 2.8 mmol) and 50 cm^3 toluene were refluxed for 1 hour until a clear solution was obtained. The solution was filtered while it was still hot. Solvent was gradually removed by evaporation under vacuum until solid product was obtained. The white powdery product was then recrystallised from toluene/hexane and kept in the fridge. Colourless needle-shaped crystals were formed. M.p. 198–200°C. Analysis: Found: C, 61.45; H, 4.62; N, 1.83%. Calculated for $C_{42}H_{34}NO_2Sn_2$: C, 61.36; H, 4.17; N, 1.70%. IR (KBr): $\nu(COO_{asym})$ 1647; $\nu(COO_{sym})$ 1349 cm^{-1}. ^1H NMR (DMSO-d_6) δ: 7.40–7.88 (m, 15H, $3C_6H_5$); 7.68 and 8.64 (2d, 4H, C_5H_4N) ppm. ^{13}C NMR

(DMSO-d_6) δ: C(O)O 167.5; C_5H_4N 123.0, 142.8, 150.2; C_6H_5 128.4, 128.8, 129.0, 135.9, 136.2, 136.5 ppm. ^{119}Sn NMR (DMSO-d_6) δ: −262 ppm.

Crystal Structure Determination

Intensity data for a crystal with dimensions 0.2 × 0.4 × 0.5 mm was measured at 298 K on a Siemens P4 diffractometer with graphite-monochromated MoK$_\alpha$ radiation, $\lambda = 0.71073$ Å. The θ-2θ scan technique was employed to measure a total of 7538 reflections in the $3.0° \leq 2\theta \leq 55.0°$ range. Corrections were applied for Lorentz and polarization effects but not for absorption. Some 4340 reflections out of the 6176 independent reflections ($R_{int} = 0.021$) satisfying the $F > 4.0\sigma(F)$ criterion of observability were used for solution and refinement. The structure was solved by direct methods and refined by a full-matrix least-squares procedure using the Siemens SHELXTL PLUS (PC version) system.[16] A weighting scheme of the form $w = [\sigma^2(F) + 0.0006F^2]^{-1}$ was used and refinement continued to final $R = 0.039$ and $R_w = 0.042$. The final difference map had peaks between −0.52 and 0.57 eÅ^{-3}.

Crystal data and refinement details are given in Table I. Fractional atomic coordinates are given in Table II and bond lengths and angles are listed in Table III. Lists of additional data, including hydrogen atom coordinates, anisotropic temperature factors and observed and calculated structure factors are available on request from the authors.

TABLE I Crystal data and refinement details for $[(C_6H_5)_3Sn(O_2CC_5H_4N) \cdot C_7H_8]_n$

Formula	$C_{31}H_{27}NO_2Sn$
Formula weight	564.2
Crystal system	Monoclinic
Space group	$P2_1/n$
a (Å)	12.3540(10)
b (Å)	15.306(2)
c (Å)	14.3080(10)
β (°)	91.000(10)
V (Å^3)	2705.1(5)
Z	4
D$_c$ (Mg m^{-3})	1.385
μ (mm^{-1})	0.971
F(000)	1144
Reflections collected	7538
Independent reflections	6176 ($R_{int} = 0.021$)
Observed reflections	4340 ($F > 4.0\sigma(F)$)
No. of parameters refined	304
R	0.039
R_w	0.042
w	$[\sigma^2(F)+0.0006F^2]^{-1}$
Largest diff. peak and hole (e Å^{-3})	0.57 and −0.52

TABLE II Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($Å^2 \times 10^3$)

	x/a	*y/b*	*z/c*	*U(eq)**
Sn	81(1)	2226(1)	1603(1)	50(1)
O(1)	1009(3)	2421(2)	361(2)	60(1)
O(2)	2119(3)	3336(2)	1120(2)	71(1)
N	3840(3)	2988(2)	−1994(2)	59(1)
C(1)	−955(3)	1358(3)	850(3)	57(1)
C(2)	−1012(6)	1419(4)	−134(4)	108(3)
C(3)	−1625(7)	831(6)	−646(5)	145(4)
C(4)	−2186(7)	183(5)	−198(6)	134(4)
C(5)	−2170(6)	125(5)	701(6)	119(3)
C(6)	−1572(5)	714(4)	1238(4)	89(2)
C(7)	−330(4)	3569(3)	1757(3)	57(1)
C(8)	−243(6)	3991(4)	2587(4)	100(3)
C(9)	−501(7)	4859(5)	2665(6)	125(3)
C(10)	−861(6)	5316(4)	1917(7)	117(3)
C(11)	−956(5)	4915(4)	1088(6)	107(3)
C(12)	−700(4)	4043(3)	1004(4)	81(2)
C(13)	1300(3)	1596(3)	2430(3)	59(1)
C(14)	2119(5)	2024(5)	2881(4)	94(2)
C(15)	2914(6)	1546(6)	3395(4)	118(3)
C(16)	2831(6)	646(6)	3451(4)	111(3)
C(17)	2026(5)	242(4)	3022(4)	92(2)
C(18)	1273(4)	691(3)	2513(4)	76(2)
C(19)	1845(3)	2914(3)	434(3)	52(1)
C(20)	2520(3)	2966(3)	−433(3)	49(1)
C(21)	3450(3)	3440(3)	−437(3)	56(1)
C(22)	4093(3)	3424(3)	−1227(3)	60(1)
C(23)	2900(4)	2561(3)	−2010(3)	70(2)
C(24)	2235(4)	2512(3)	−1239(3)	64(1)
C(25)	4976(16)	1615(10)	552(13)	507(25)
C(26)	5330	2280	813	295(13)
C(27)	4822(6)	2536(7)	1633(7)	197(9)
C(28)	5118	3316	2071	250(16)
C(29)	5920	3841	1690	195(9)
C(30)	6428	3586	870	350(17)
C(31)	6133	2805	432	245(10)

*Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

RESULTS AND DISCUSSION

The molecular structure of the asymmetric unit of triphenyltin(IV) 4-pyridinecarboxylate is depicted in Figure 1. The geometry about the tin atom is that of a slightly distorted trigonal bipyramid, with the *ipso*-carbons of the phenyl groups defining the trigonal plane such that the sum of trigonal C–Sn–C angles is 358.4°. The apical positions are occupied by the O(1) atom of the carboxylate group and the N′ atom of the neighbouring pyridyl ring (where N′ is related by the symmetry operation $-0.5+x$, $0.5-y$, $0.5+z$); the O(1)–Sn–N′ angle is 175.5(1)°. This arrangement demands

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

Sn–O(1)	2.152(3)	Sn–C(1)	2.125(4)
Sn–C(7)	2.129(4)	Sn–C(13)	2.130(4)
Sn–N′	2.568(4)	Sn–O(2)	3.124(3)
O(2)–C(19)	1.217(5)	O(1)–C(19)	1.283(5)
N–C(23)	1.332(6)	N–C(22)	1.317(5)
C(1)–C(2)	1.411(7)	N–Sn′	2.568(4)
C(2)–C(3)	1.379(11)	C(1)–C(6)	1.370(7)
C(4)–C(5)	1.289(12)	C(3)–C(4)	1.375(12)
C(7)–C(8)	1.355(7)	C(5)–C(6)	1.389(9)
C(8)–C(9)	1.372(10)	C(7)–C(12)	1.370(7)
C(10)–C(11)	1.338(12)	C(9)–C(10)	1.347(12)
C(13)–C(14)	1.359(8)	C(11)–C(12)	1.378(8)
C(14)–C(15)	1.419(10)	C(13)–C(18)	1.391(7)
C(16)–C(17)	1.314(10)	C(15)–C(16)	1.384(14)
C(19)–C(20)	1.509(6)	C(17)–C(18)	1.358(8)
C(20)–C(24)	1.387(6)	C(20)–C(21)	1.358(6)
C(23)–C(24)	1.389(7)	C(21)–C(22)	1.394(6)
C(25)–C(26)	1.167(19)		
O(1)–Sn–C(1)	89.5(1)	O(1)–Sn–C(7)	94.8(1)
C(1)–Sn–C(7)	120.9(2)	O(1)–Sn–C(13)	98.0(1)
C(1)–Sn–C(13)	114.3(2)	C(7)–Sn–C(13)	123.2(2)
O(1)–Sn–N′	175.5(1)	C(1)–Sn–N′	87.4(1)
C(7)–Sn–N′	84.0(1)	C(13)–Sn–N′	86.2(1)
Sn–O(1)–C(19)	117.0(2)	C(22)–N–C(23)	117.2(4)
C(22)–N–Sn′	125.4(3)	C(23)–N–Sn′	117.1(3)
Sn–C(1)–C(2)	118.9(4)	Sn–C(1)–C(6)	125.4(4)
C(2)–C(1)–C(6)	115.7(5)	C(1)–C(2)–C(3)	120.3(6)
C(2)–C(3)–C(4)	119.9(7)	C(3)–C(4)–C(5)	121.0(8)
C(4)–C(5)–C(6)	120.5(7)	C(1)–C(6)–C(5)	122.5(6)
Sn–C(7)–C(8)	122.4(4)	Sn–C(7)–C(12)	120.5(3)
C(8)–C(7)–C(12)	117.2(4)	C(7)–C(8)–C(9)	121.2(6)
C(8)–C(9)–C(10)	120.8(7)	C(9)–C(10)–C(11)	119.3(6)
C(10)–C(11)–C(12)	120.3(7)	C(7)–C(12)–C(11)	121.2(6)
Sn–C(13)–C(14)	124.0(4)	Sn–C(13)–C(18)	118.8(3)
C(14)–C(13)–C(18)	117.3(5)	C(13)–C(14)–C(15)	120.0(6)
C(14)–C(15)–C(16)	119.5(7)	C(15)–C(16)–C(17)	119.8(7)
C(16)–C(17)–C(18)	121.2(7)	C(13)–C(18)–C(17)	122.2(5)
O(1)–C(19)–O(2)	126.2(4)	O(1)–C(19)–C(20)	114.8(3)
O(2)–C(19)–C(20)	119.0(4)	C(19)–C(20)–C(21)	120.8(3)
C(19)–C(20)–C(24)	121.4(4)	C(21)–C(20)–C(24)	117.8(4)
C(20)–C(21)–C(22)	119.3(4)	N–C(22)–C(21)	123.6(4)
N–C(23)–C(24)	122.7(4)	C(20)–C(24)–C(23)	119.2(4)
C(25)–C(26)–C(27)	110.0(10)	C(25)–C(26)–C(31)	130.0(10)

bidentate bridging from the carboxylate ligand leading to a chain-like polymeric structure with a nearly linear O–Sn–N backbone propagating along the *a* axis (Figure 2). It can be seen that (Figure 1) toluene molecules are present in the crystal lattice of the complex in a 1 : 1 ratio. The toluene molecules do not participate in any significant interaction to the tin atom, but may contribute to the stabilization of the crystal lattice.

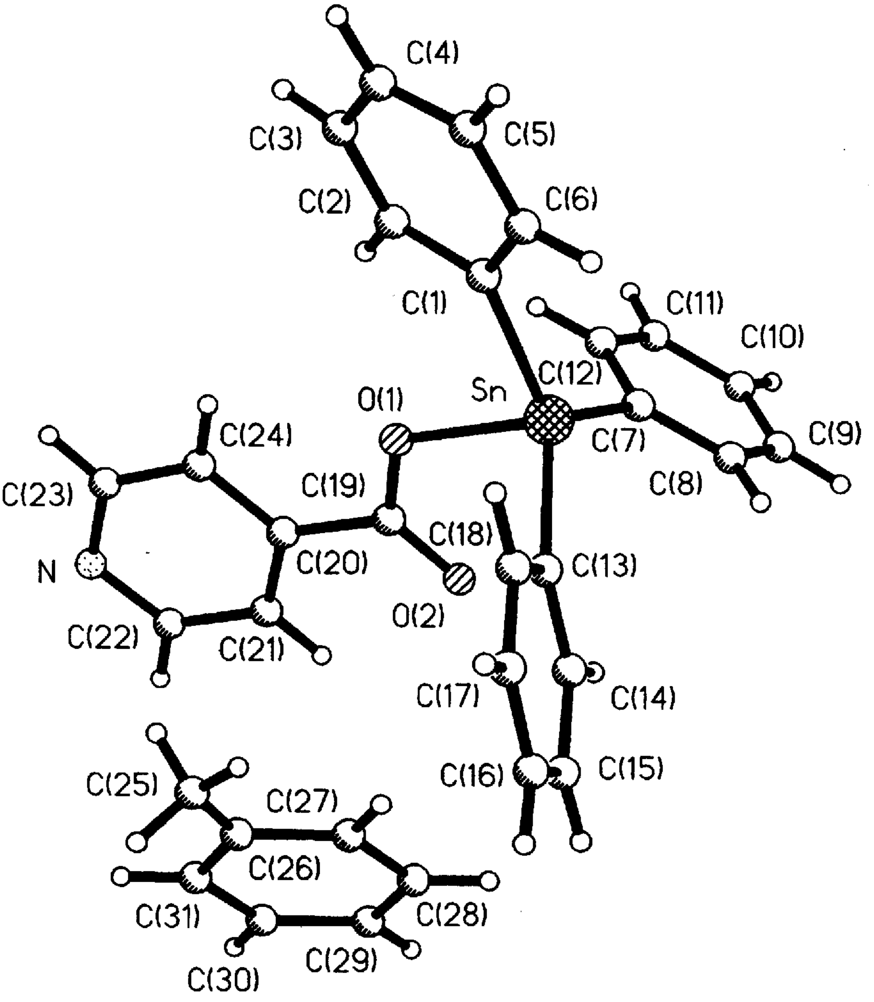

FIGURE 1 Asymmetric unit of $[(C_6H_5)_3Sn(O_2CC_5H_4N)\cdot C_7H_8]$ showing the labelling scheme used.

The structure of the title triorganotin carboxylate resembles those of the *trans*-R_3SnO_2 structural type.[1] However, the two donor atoms of the 4-pyridinecarboxylate ligand are the O(1) and the pyridine N atoms as in the structure of triphenyltin(IV) 3-pyridinecarboxylate.[14] The Sn–N′ bond of the title compound is 2.568(4) Å and is similar to that of the triphenyltin 3-pyridinecarboxylate (2.568(7) Å). Nevertheless, the Sn–O(1) bond distance of the title compound (2.152(3) Å) is greater than that of the latter complex

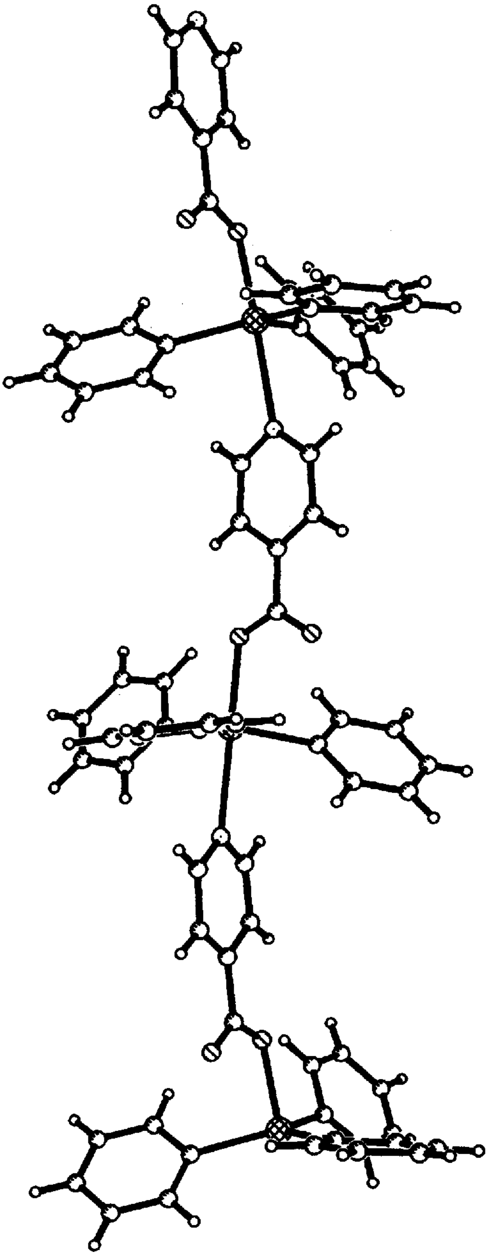

FIGURE 2 A polymeric chain of triphenyltin(IV) 4-pyridinecarboxylate propagating along the *a* axis.

(2.137(6) Å). A helix is better defined for those complexes where the difference between the Sn–O and Sn–N bond lengths is small, whereas for larger differences, it has been reported that the helix becomes stretched into an S configuration.[3,17] It has also been reported that in triphenyltin 3-pyridinecarboxylate, the difference between its Sn–O and Sn–N′ bond distances is 0.431 Å, thus having a stretched helical nitrogen-bridged polymeric configuration.[14] The one-dimensional structure of the polymer chain of the title compound can be described as a helix since the difference between the Sn–O and Sn–N′ distances is smaller (0.416 Å).

The intramolecular Sn···O(2) contact is 3.124(3) Å, and the C(19)–O(1) bond seems to be rotated to bring the O(2) atom into juxtaposition with the tin atom. The O(2) atom does not form a significant interaction with the tin atom. However, the close proximity of the O(2) atom influences the coordination geometry about the tin atom by opening up the C(7)–Sn–C(13) angle to 123.2(2)°.

The average Sn–C(Ph) distance of 2.128(4) Å is within the range [2.105(5)–2.160(2) Å] of Sn–C(Ph) distances reported in the literature.[18] All the C–C bond lengths of the phenyl groups appear to be normal.

The repeat distance in polymeric triorganotin carboxylates has been used to distinguish the various types of bridging in these structures.[19] The carboxylate-bridged triorganotin carboxylate polymers, those best described as the *trans*-R_3SnO_2 type, give the average repeat distance of 5.19 ± 0.21 Å.[19] The unit cell constants for the title compound are shown in Table I where $a = 12.3540(10)$, $b = 15.306(2)$ and $c = 14.3080(10)$ Å and none of these dimensions divided by 2 or 4 yields a value close to 5.19 Å. As a result, a bridging interaction by the pyridine-N atom is inferred, and in fact, demonstrated by the full X-ray crystal structure determination.

The assignment of IR bands has been made by comparison between the IR spectra of the complex and the free acid and its sodium salt. The IR spectra show that the band assignable to the asymmetrical stretching of COO for 4-pyridinecarboxylic acid is shifted from 1710 cm^{-1} to 1647 cm^{-1} whereas the band for symmetrical COO stretching is shifted towards higher frequency, from 1305 to 1349 cm^{-1}, upon complex formation. The shifts in the $\nu(\mathrm{COO_{asym}})$ and $\nu(\mathrm{COO_{sym}})$ bands indicate that the carboxylate group of the free acid is coordinated to the tin atom.[20,21] The absence of broad band around 2500–2800 cm^{-1} due to OH absorption of COOH further supports the coordination of the carboxylate group after deprotonation. IR data for the title compound also imply the presence of the unidentate carboxylate group with $\Delta\nu$ [$\Delta\nu = \nu(\mathrm{COO_{asym}}) - \nu(\mathrm{COO_{sym}})$] of 298 cm^{-1}, which lies between the value of the free acid (405 cm^{-1}) and of the sodium salt

(176 cm^{-1}).[22,23] The $\nu(COO_{asym})$ absorption of the title complex is similar to that of tri-*n*-butyltin(IV) 3- and 4-pyridinecarboxylates, which shows that pentacoordination does not occur through the carbonyl oxygen atom but instead involves the nitrogen atom of the pyridyl ring.[15] The absorption bands of Sn–O, Sn–N and Sn–C cannot be observed in the spectrum of the complex as they lie below 450 cm^{-1}.[23–25]

^{1}H, ^{13}C and ^{119}Sn NMR spectra of the title complex have been recorded in DMSO-d_6 solution. The ^{1}H NMR spectrum shows the expected integration and peak multiplicities. Signals for protons of the pyridyl ring of the 4-pyridinecarboxylate ligand overlap with the aromatic protons of the phenyl group and are thus difficult to distinguish. However, after comparing with the ^{1}H NMR spectrum of the ligand, two doublets observable at δ 7.68 and 8.64 ppm are ascribable to the pyridyl ring and the signals from the phenyl group appear as complex multiplets in the region δ 7.40–7.88 ppm.

The ^{13}C NMR spectrum for the compound shows only one set of Sn–Ph and ligand carbon resonances. Once again, it is hard to distinguish the chemical shifts of the carbon resonances of the phenyl group and the pyridyl ring. Furthermore, some of the signals are obscured due to overlapping. Signals at δ 123.0, 142.8 and 150.2 ppm are for the carbons of the pyridyl ring while the aromatic phenyl carbons appear at δ 128.4, 128.8, 129.0, 135.9, 136.2, 136.5 ppm by reference to the ^{13}C NMR spectrum of the ligand. The carbonyl carbon absorbs further downfield (δ 167.5 ppm) in the spectrum of the complex.

The ^{119}Sn chemical shift at δ -262 ppm is consistent with a trigonal bipyramidal geometry around a five-coordinate tin atom.[24] This of course is consistent with the X-ray crystal structure determination.

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