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Synthesis and Characterization of Trialkyltin Esters of Pyridinecarboxylic Acids and Crystal Structure of $[^{n}\mathrm{Bu_3SnO_2CC_5H_4N\text{-}2}]_\infty$

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ABSTRACT: *Reactions of trialkyltin oxides with 2-, 3-, and 4-pyridinecarboxylic acids in 1:1 stoichiometry yield 10 corressponding trialkyltin esters* $R_3SnO_2CC_5H_4N$. *All compounds are characterized by elemental analysis, IR,* ^{1}H, ^{113}C, *and* ^{119}Sn *NMR. The crystal structure of tributyltin ester of 2-pyridinecarboxylic acid is determined by single crystal X-ray diffraction. In this compound, the tin atom is rendered five-coordinate in a trigonal bipyramidal structure by coordinating with 2-pyridinecarboxylate group. The resulting structure is a linear polymer containing Sn—O bonds (0.2278(5) and 0.2308(6) nm).* © 2004 Wiley Periodicals, Inc. Heteroatom Chem 15:524–529, 2004; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20057

INTRODUCTION

Recently, organotin(IV) compounds with carboxyl groups have received increased attention owing to their antitumor activities [1–6]. In general, the biological activity of organotin compounds is influenced greatly by the structure of the molecule and the coordination number of the tin atom(s) [7–9]. Studies on organotin compounds containing carboxylate ligands with additional donor atom (e.g. N, O, or S) which are available for coordinating to tin atom have revealed that new structural types may lead to different activity [10,11]. As an extension of our studies of organotin esters of carboxylic acid with additional donor atoms residing on the carboxylate ligand [12,13], we have synthesized and structurally characterized a series of trialkytin compounds derived from 2-, 3-, and 4-pyridinecarboxylic acids and determined the crystal structure of $[^{n}\mathrm{Bu_3SnO_2CC_5H_4N\text{-}2}]_\infty$. And the results of these studies are reported herein.

EXPERIMENTAL

General Procedures

All reactants were reagent grade. Infrared spectra were recorded on a Nicolet-460 spectrophotometer using KBr disks. ^{1}H, ^{113}C, and ^{119}Sn NMR spectra were obtained on Mercury Plus-400 NMR spectrometer; chemical shifts were given in parts per million relative to Me_4Si and Me_4Sn in $CDCl_3$ solvent. Elemental analyses were performed with a PE-2400 II elemental apparatus. Tin was estimated as SnO_2. X-ray measurements were made on a Bruker Smart-1000 CCD diffractometer with graphite-monochromated Mo Kα (0.071073 nm) radiation.

Synthesis of Trialkyltin Esters of Pyridinecarboxylic Acid

Pyridinecarboxylic acids (1.2 mmol) were added to a benzene solution of $(R_3Sn)_2O$ (1.0 mmol). Then

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the mixture was refluxed for 5 h with water formed during the reaction being removed azeotropically with a Dean and Stark apparatus. The clear solution obtained after filtration was evaporated in vacuum to give a white solid. The products were recrystallized from acetone-hexane to give a colorless crystal.

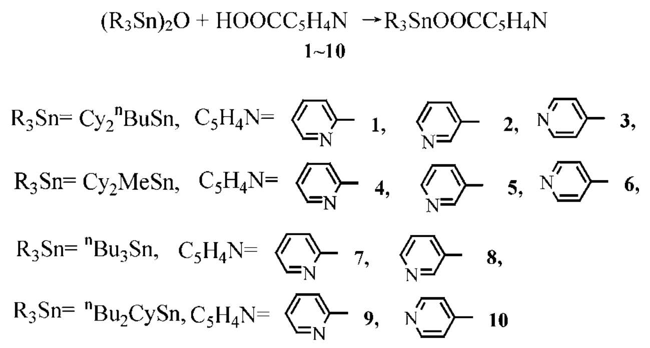
Crystallographic Measurements of [^{n}Bu$_3$SnO$_2$CC$_5$H$_4$N-2]$_\infty$

The colorless crystal having approximate dimension of 0.22 mm × 0.10 mm × 0.08 mm was mounted on a glass capillary. All measurements were made on a Bruker Smart 1000 CCD diffractometer with graphite-monochromated Mo Kα (0.071073 nm) radiation. The data were collected at room temperature (298 ± 2 K) using the ϕ-ω scan technique. The crystal structure belongs to monoclinic with space group $C2/c$, $a = 2.6503(5)$ nm, $b = 1.0988(2)$ nm $c = 1.9532(4)$ nm, $\beta = 131.63(3)^\circ$, $V = 4.2515(15)$ nm^3, $Z = 8$, $D_c = 1.288$ g/cm^3, $\mu = 1.208$ mm^{-1}, and $F(000) = 1696$. The structure was solved by direct method and differential Fourier map using SHELXL-97 program, and refined by full-matrix least-squares on F^2. All nonhydrogen atoms were refined anisotropically. Position of hydrogen atoms was calculated and refined isotropically. The weighting scheme was $w = 1/[s^2(F_o^2) + (0.0403P)^2 + 0.0000P]$, $p = (F_o^2 + 2F_c^2)/3$. The refinement was converged to the final $R = 0.0493$, $wR = 0.0919$. In the final difference map, the residuals are 0.487 and -0.254 e/Å^3 respectively.

RESULTS AND DISCUSSION

Physical Properties

Physical data for compounds **1–10** are listed in Table 1. All compounds are colorless crystals. They are soluble in many organic solvents as CCl$_4$, CHCl$_3$, C$_6$H$_6$, (CH$_3$)$_2$CO, and THF but are insoluble in hexane, petroleum ether, and water.

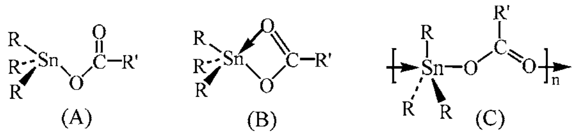


SCHEME 1

Analysis of Spectroscopy

IR Spectra. The characteristic IR spectral data of the compounds **1–10** are shown in Table 2. The assignment of IR bands of these compounds has been determined by comparison with the IR spectra related organotin compounds, carboxylates, and (R$_3$Sn)$_2$O [1,8]. It is to note that the difference $\Delta\nu$ between ν^{as}(COO) and ν^{s}(COO) is important because these frequencies can be used to determine the type of bonding between metal and carboxyl [1,12,13]. Generally triorganotin esters of carboxylic acid adopt three types of structures in the solid state (as shown in Scheme 2).

The magnitude of $\Delta\nu[\nu^{as}(\mathrm{COO})-\nu^{s}(\mathrm{COO})]$ of 176 cm^{-1} and 186 cm^{-1} for compounds **7** and **9** is approximately same as those for the corresponding sodium of carboxylates, which indicates the presence of bidentate carboxylate groups [1–4]. So it is impossible for these compounds to be structure **A**. Otherwise, IR spectra of these compounds in CCl$_4$ are to be studied to find out whether the ligand linkage of C=O bond to tin atom is intramolecular or intermolecular [1,14]. Because in solution the intermolecular bond of the five-coordinated organotin polymer that belongs to structure **C** is destroyed, the polymeric structure is decomposed into monomolecule and as a result, the $\Delta\nu$ value increases greatly [14,15]. However $\Delta\nu$ values for compounds with structures **B** and **A** do not change obviously when they were determined in solvent [14]. The results showed that the magnitude of $\Delta\nu$ 299 cm^{-1} and 315 cm^{-1} in CCl$_4$ is much greater than that in KBr for compounds **7** and **9**. The fact revealed that the structure of these compounds might be structure **C**, not be structure **B**. The magnitude of 290–310 cm^{-1} for compounds **1–6, 8**, and **10** strongly indicates the unbidentate chelating of the carboxylate groups, and the results in KBr are similar to those in CCl$_4$. But the structures of these compounds are different from structures **A** and **C**. One obvious feature of the IR spectra in the compounds **1–10** at 471–492 cm^{-1} is the similarity of stretching band arising from Sn—N [15]. Because the intermolecular bond of Sn ← N will break in solvent,



SCHEME 2

TABLE 1 Physical and Analytical Data of Compounds **1–10**

Compound	*Yield (%)*	*mp (°C)*	*Found (Calcd)%*			
			C	*H*	*N*	*Sn*
1	80.5	73–75	56.77(56.92)	7.55(7.60)	2.92(3.02)	25.71(25.57)
2	88.4	102–103	56.85(56.92)	7.83(7.60)	2.99(3.02)	25.69(25.57)
3	85.8	136–137	57.11(56.92)	7.70(7.60)	3.02(3.02)	25.48(25.57)
4	84.4	80–82	54.29(54.06)	7.10(6.92)	3.38(3.32)	28.05(28.12)
5	77.8	99–100	53.87(54.06)	6.84(6.92)	3.40(3.32)	28.03(28.12)
6	84.6	127–129	54.34(54.06)	6.97(6.92)	3.44(3.32)	28.17(28.12)
7	77.8	120–122	52.73(52.67)	7.52(7.76)	3.47(3.40)	28.53(28.80)
8	87.6	152–154	52.65(52.67)	7.69(7.76)	3.49(3.40)	29.05(28.80)
9	79.1	63–65	54.69(54.82)	7.46(7.59)	3.27(3.20)	27.33(27.09)
10	80.4	111–112	55.01(54.82)	7.64(7.59)	3.28(3.20)	27.19(27.09)

while the intramolecular bond of Sn ← N is not influenced by solvent [14], whether Sn ← N of organotin compound is intermolecular or intramolecular may be deduced. It is shown that N hetero atom on the pyridine group in carboxylate group coordinates to tin atom [15]. In CCl_4 solution, this band is not observed in compounds **2, 3, 5, 6, 8**, and **10**. This indicates that the Sn ← N bond was broken up for the compounds in CCl_4 solution, Thus it is proved that compounds **2, 3, 5, 6, 8**, and **10** contain intermolecular bond of Sn ← N and exists as a polymer [13,15]. However, this difference is not observed for compounds **1** and **4** ($\nu_{\mathrm{Sn}\leftarrow\mathrm{N}}$ 485 cm^{-1}] and 480 cm^{-1} in CCl_4). Since the intramolecular coordination bond is basically not influenced by solvent [14], the bond Sn ← N existing in compounds **1** and **4** with structure **F** (see Scheme 3) can also be observed in solvent yet. So it is possible for compounds **2, 3, 5, 6, 8**, and **10** to be structure **D** or **E**, and compound **1** and **4** to be structure **F** [15]. Furthermore the structures of the compounds **7** and **9** are slightly different from structures **C** and **F**. One obvious feature of the IR spectra in two compounds at 471 and 475 cm^{-1} also is the similarity of stretching band arising from Sn–N [15]. So it is possible for compounds **7** and **9** to may be structure **G**.

^{1}H, ^{13}C, and ^{119}Sn NMR Spectra. The ^{1}H, ^{13}C, and ^{119}Sn NMR spectra of compounds **1–10** are given in Tables 3 and 4.

The chemical shifts of the protons of pyridine ring of compounds **1–10** exhibit signals at 7.20–9.15 ppm as multiplet. The chemical shifts of the protons of pyridine ring obviously increase in comparison with that corresponding free heteroarmatic acid. This finding may be consistent with previous conclusion, which suggests that pyridine type N atoms, present in the carboxylate R′ group, coordinate to Sn [14,15].

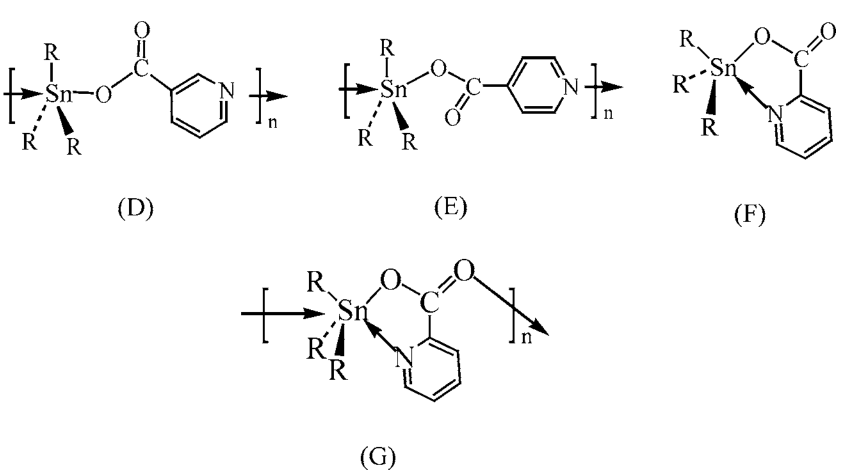
In the ^{13}C NMR spectra of compounds **1–10**, chemical shifts are similar to the free ligand. The carbons show a slightly downfield shift of all carbon resonances compared with the free ligand in compounds **1, 4, 7**, and **9**. This finding may be consistent with pyridine type N atoms, present in the carboxylate R′ group, coordinate to Sn in solvent [14,15].

The ^{119}Sn NMR spectroscopic data are very useful for determining the coordination number of tin,

TABLE 2 IR Data (cm^{-1}) of Compounds **1–10**

Compound	ν^{as}*(COO)*	ν^{s}*(COO)*	$\Delta\nu$	ν*(Sn—C)*	ν*(Sn—N)*	ν*(Sn—O)*
1	1645s(1649)	1352s(1348)	293(301)	554w	492w(485)	453m
2	1640s(1644)	1341s(1344)	299(300)	562w	481w	442w
3	1644s(1643)	1350s(1346)	294(297)	552m	478w	452m
4	1650s(1652)	1358s(1362)	292(290)	552w	487w(480)	446m
5	1638s(1647)	1342s(1337)	296(310)	558w	490w	452w
6	1648s(1647)	1358s(1354)	310(293)	548m	481w	460m
7	1571s(1642)	1395s(1343)	176(299)	545w	471w(484)	447w
8	1640s(1647)	1350s(1344)	290(303)	555w	487w	449m
9	1588s(1655)	1402s(1340)	186(315)	540w	475w(482)	450m
10	1638s(1649)	1365s(1357)	273(292)	550m	482w	448m

The parentheses give the vibration of compounds in CCl_4.



(D) (E) (F)

(G)

SCHEME 3

its molecular geometry, and stereochemistry. In the dialkyltin esters, four-coordinate tin has δ(^{119}Sn) values ranging from about +200 to −60 ppm, five-coordinate tin from −90 to −190 ppm, six-coordinate tin from −210 to −400 ppm, and seven-coordinate tin from −450 to −460 ppm [16,17].

The ^{119}Sn NMR data for compounds **2, 3, 5, 6, 8,** and **10** indicate that a signal appearing at −25.4 to −42.3 ppm suggests that the tin atoms exhibit four-coordinate in solution state. And in compounds **1, 4, 7,** and **9**, the ^{119}Sn chemical shift values are found to be in the range of −204.7 to −210.3 ppm. The appearance of chemical shift values in this region indicates five-coordination environment around the central tin atoms in solution state [16,17].

*X-ray Crystal Structure of [^{n}Bu$_3$SnO$_2$CC$_5$H$_4$N-2]$_\infty$(**7**)*

The selected bond distances and angles of compound **7** are listed in Table 5. The molecular structure and One-dimensional chain network are shown in Figs. 1 and 2.

Tributyltin ester of 2-pyridinyl carboxylic acid possesses an unequivocally polymeric structure. Each tin atom is rendered five-coordinate by coordination of the "free" oxygen of 2-pyridinyl carboxyltate group from an adjacent molecule. The coordination at the tin atom is distorted trigonal bipyramidal, surrounded axially by two oxygen atoms and equatorially by three α-carbon atoms of the

TABLE 3 ^{1}H NMR Data (ppm) for Compounds **1–10**

No	*^{1}H NMR*	*^{119}Sn NMR*
1	8.42 (1H,br, 6-Py-H), 8.15 (1H, br, 3-Py-H), 7.84 (1H, br, 4-Py-H), 7.37 (1H, br, 5-Py-H), 1.18–2.15 (28H, m, SnCH$_2$CH$_2$CH$_2$, SnCy-H), 0.98 (3H, t, J = 6.8 Hz, CH$_3$)	−102.4
2	9.08 (1H, s, 2-Py-H), 8.75 (1H, br, 6-Py-H), 8.15 (1H, br, 5-Py-H), 7.30 (1H, br, 3-Py-H), 1.20–2.18 (28H, m, SnCH$_2$CH$_2$CH$_2$, SnCy-H), 0.95 (3H, t, J = 7.0 Hz, CH$_3$)	−42.3
3	8.75 (2H, br, 2,6-Py-H), 7.68 (2H, br, 3,5-Py-H), 1.19–2.20 (28H, m, SnCH$_2$CH$_2$CH$_2$, SnCy-H), 0.99 (3H, t, J = 6.8 Hz, CH$_3$)	−40.2
4	8.44 (1H,br, 6-Py-H), 8.20 (1H, br, 3-Py-H), 7.82 (1H, br, 4-Py-H), 7.35 (1H, br, 5-Py-H), 1.26–2.01 (22H, m, SnCy-H), 0.38 (3H, s, $J_{\mathrm{Sn-H}}$ = 45.4 Hz, CH$_3$)	−95.5
5	9.12 (1H, br, 2-Py-H), 8.80 (1H, br, 6-Py-H), 8.21 (1H, br, 5-Py-H), 7.28 (1H, br, 3-Py-H), 1.30–1.97 (22H, m, SnCy-H), 0.36 (3H, s, $J_{\mathrm{Sn-H}}$ = 44.2 Hz, CH$_3$)	−32.7
6	8.81 (2H, br, 2,6-Py-H), 7.64 (2H, br, 3,5-Py-H), 1.28–2.04(22H, m, SnCy-H), 0.37 (3H, s, $J_{\mathrm{Sn-H}}$ = 45.8 Hz, CH$_3$)	−30.5
7	8.72 (1H, br, 6-Py-H), 8.13 (1H, br, 3-Py-H), 7.80 (1H, br, 4-Py-H), 7.42 (1H, br, 5-Py-H), 1.16–1.80 (18H, m, SnCH$_2$CH$_2$CH$_2$), 0.92 (3H, t, J = 7.0 Hz, CH$_3$)	−108.3
8	9.15 (1H, br, 2-Py-H), 8.78 (1H, br, 6-Py-H), 8.17 (1H, br, 5-Py-H), 7.34 (1H, br, 3-Py-H), 1.20–1.75 (18H, m, SnCH$_2$CH$_2$CH$_2$), 0.90 (3H, t, J = 7.0 Hz, CH$_3$)	−25.4
9	8.80 (1H,br, 6-Py-H), 8.12 (1H, br, 3-Py-H), 7.85 (1H, br, 4-Py-H), 7.41 (1H, br, 5-Py-H), 1.23–1.99 (23H, m, SnCH$_2$CH$_2$CH$_2$, SnCy-H), 0.89 (3H, t, J = 7.0 Hz, CH$_3$)	−114.6
10	8.70 (2H, br, 2,6-Py-H), 7.66 (2H, br, 3,5-Py-H), 1.20–2.02 (23H, m, SnCH$_2$CH$_2$CH$_2$,SnCy-H), 0.88 (3H, t, J = 7.0 Hz, CH$_3$)	−34.2

TABLE 4 ^{13}C NMR Data (ppm) for Compounds **1–10**

	Cy–C				*Bu–C or Me–C*					
No	*Cα*	*Cβ*	*Cγ*	*Cδ*	*C₁*	*C₂*	*C₃*	*C₄*	*C=O*	*Py–C*
1	34.54	30.78	28.90	26.85	15.45	28.10	27.61	13.75	179.12	126.45, 128.87, 142.01, 147.49, 148.83
2	34.12	30.23	27.99	26.76	15.01	27.63	27.25	13.43	172.55	120.81, 129.02, 138.36, 148.98, 160.24
3	34.35	30.16	28.06	26.77	15.34	27.89	27.56	13.57	173.82	123.56, 128.98, 140.14, 148.55, 150.48
4	34.72	30.55	28.34	26.70	−8.12				177.24	125.90, 128.38, 141.91, 147.23, 148.50
5	31.98	29.76	27.98	26.32	−6.54				168.72	120.56, 128.75, 138.52, 149.33, 159.60
6	32.12	30.11	28.21	26.55	−6.88				172.67	123.75, 128.54, 140.37, 148.01, 151.11
7					16.82	27.81	26.86	13.77	172.98	126.24, 128.13, 141.57, 147.05, 148.09
8					16.01	27.54	26.75	13.20	171.36	120.27, 128.65, 138.80, 150.32, 159.47
9	34.12	30.38	28.54	26.57	15.79	27.78	27.00	13.56	173.32	126.70, 128.55, 141.38, 147.43, 148.36
10	33.91	30.23	28.37	26.60	15.46	27.34	27.06	13.72	171.45	123.28, 128.90, 141.23, 148.47, 151.04

n-butyl groups. The structure is similar to the compound $Cy_3SnO_2CC_3H_7$ [18] and $(Ph_3CH_2)_3SnO_2CCH_3$ [19], but is different from complexes triphenyltin ester of 3-pyridinecarboxylic acid [20], triphenyltin ester of 4-pyridinecarboxylic acid [21], tributylin ester of 4-pyridinecarboxylic acid, tribenzyltin esters of 4-pyridinecarboxylic acid [22], and tri(*o*-fluorobenzyl)tin esters of 4-pyridinecarboxylic acid and 3-pyridinecarboxylic acid [23].

The intramolecular Sn(1)—O(1) bond distance of 0.2278(5) nm is longer than that those of $Cy_3SnO_2CC_3H_7$ (0.2142 nm), $(Ph_3CH_2)_3SnO_2CCH_3$ (0.214 nm) [19], $(PhCH_2)_3SnOO_2CC_5H_4N$-4 (0.2142 nm) [22], and $Me_3SnO_2CC_5H_4N{\cdot}H_2O$ (0.218 nm and 0.221 nm) [24]. The intermolecular Sn(1)—O(2a) distance of 0.2308(6) nm is shorter than that in $(Ph_3CH_2)_3SnO_2CCH_3$ (0.265 nm) and $(CH_3)_3SnO_2CC_5H_4N{\cdot}H_2O$ (0.243 nm). The Sn(1)—N(1A) distance 0.3120(5) nm is much greater than the sum of the covalent radii of Sn and N (0.215 nm), but is considerably less than the sum of the van der Waals radii (0.375 nm) [25] and should be considered as weak bonding interaction. In this connection, it is relevant to note the Sn—N bond distances found in other crystal structures of organotin compounds containing the pyridine carboxylate ligand. In the polymer $[Me_2SnCl(2\text{-pic})]_n$ the two unique Sn—N bond distances are 0.250(3) nm and 0.247(2) nm [26], but in the polymer $[Me_2Sn(2\text{-pic})_2]_n$ the two Sn—N bond distances are 0.2507(4) nm and 0.2477(4) nm [10], and in dicarboxylato tetraorganostannoxane $\{[^nBu_2Sn(2\text{-pic})]_2O\}_2$ the two Sn—N bond distances are 0.2550(5) nm and 0.3150(5) nm [7]. In linear polymer $(PhCH_2)_3SnOO_2CC_5H_4N$-4, the Sn—N distance is 0.2563 nm [22].

In compound **7**, the distortions from true trigonal bipyramidal symmetry are reflected in the interatomic angles. The angles of C(7)—Sn(1)—O(1), 88.5(3); C(11)—Sn(1)—O(1), 86.2(3); C(15)—Sn(1)—O(1), 90.5(3); C(7)—Sn(1)—O(2A), 97.0(3); C(11)—Sn(1)—O(2A), 83.2(3); C(15)—Sn(1)—O(2A), 93.4(3)° all are not 90°, and only one approximates to 90°. The angle O(1)—Sn(1)—O(2A) (169.40(19)°) is larger than that angle (168°) in compound $(PhCH_2)_3SnO_2CCH_3$ [19], but is less than the mean value of 174.6° in compound $(CH_3)_3SnO_2CC_5H_4N{\cdot}H_2O$ [24], this shows that the atoms O(1), Sn(1), and O(2A) are nearly linear.

SUPPLEMENTARY MATERIAL

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 236918 for compound **7**. Copies of this information may be obtained from

TABLE 5 Selected Bond Distances (nm) and Angles (°) for Compound **7**

Sn(1)–C(7)	0.2064(12)	Sn(1)–O(2A)	0.2308(6)	O(2A)–C(1A)	0.1226(9)
Sn(1)–C(15)	0.2072(11)	O(1)–C(1)	0.1254(9)	C(1)–C(2)	0.1489(11)
Sn(1)–C(11)	0.2135(9)	O(2)–C(1)	0.1226(9)	C(1A)–C(2A)	0.1498(11)
Sn(1)–O(1)	0.2278(5)	O(1A)–C(1A)	0.1254(9)	Sn(1)–N(1A)	0.3120(5)
C(7)–Sn(1)–C(15)	127.7(4)	C(11)–Sn(1)–O(1)	86.2(3)	C(1)–O(1)–Sn(1)	128.1(6)
C(7)–Sn(1)–C(11)	115.9(4)	C(7)–Sn(1)–O(2A)	97.0(3)	O(2)–C(1)–O(1)	125.3(9)
C(15)–Sn(1)–C(11)	116.1(4)	C(15)–Sn(1)–O(2A)	93.4(3)	O(2)–C(1)–C(2)	118.9(9)
C(7)–Sn(1)–O(1)	88.5(3)	C(11)–Sn(1)–O(2A)	83.2(3)	O(1)–C(1)–C(2)	115.8(9)
C(15)–Sn(1)–O(1)	90.5(3)	O(1)–Sn(1)–O(2A)	169.40(19)	N(1)–C(2)–C(1)	117.0(10)

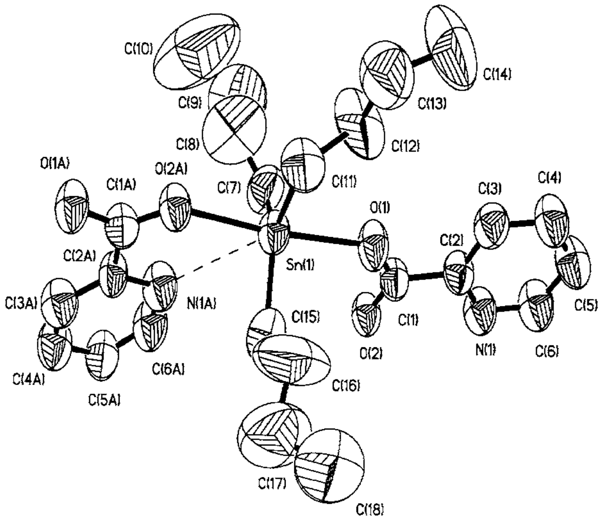


FIGURE 1 The molecular structure of compound **7**.

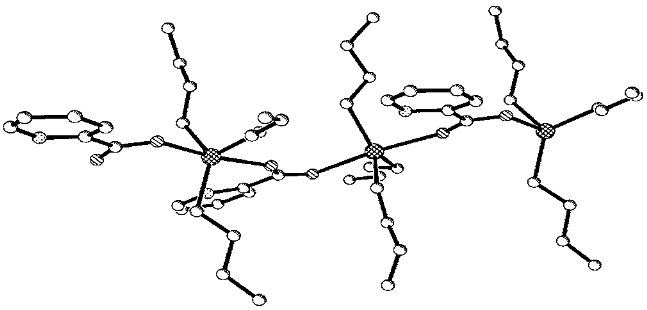


FIGURE 2 One-dimensional chain network of compound **7**.

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