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Synthesis and characterization of organotin(IV) compounds with Schiff base of *o*-vanillin-2-thiophenoylhydrazone

Note

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Abstract

Seven Schiff base adducts of organotin(IV), RSnLCl₂, which L is *o*-vanillin-2-thiophenoylhydrazone, and R is n-C₄H₉ (1), Me (2), Ph (3), and [R₂SnL], which L is *o*-vanillin-2-thiophenoylhydrazone, R is n-C₄H₉ (4), Me (5), Ph (6), PhCH₂ (7) have been synthesized. Those products were characterized by elemental analysis, IR, ¹H, ¹³C and ¹¹⁹Sn NMR spectra. The crystal and molecular structures of compounds 1, 4, and 6 have been determined by X-ray single crystal diffraction. In the crystal of compound 1 the tin atom is rendered six-coordinate in a distorted octahedral configuration by coordinating with the N atom of the Schiff base ligand, in compounds 4 and 6 the central tin atoms are five-coordinate in distorted trigonal-bipyramidal geometry and the comparison of the IR spectra reveal that disappearance of the bands assigned to carboxyl unambiguously conforms the ligand coordinate with the tin atom in enol form. © 2006 Elsevier B.V. All rights reserved.

Keywords: Organotin(IV); o-Vanillin-2-thiophenoylhydrazone; Crystal structures

1. Introduction

Organotin(IV) complexes have been the subject of interest for some time because of their biomedical and commercial application [1]. In recent years, there have been more and more reports on the synthesis, anti-tumor activities and structural elucidations of various diorganotin derivatives of carboxylic acid [2–5]. Increasing attention has also been devoted to the organotin(IV) complexes with Schiff base ligand in view of their special anti-tumour activities [6–14]. In addition to their anti-tumor activities, organotin(IV) complexes with Schiff bases present an interesting variety of structural possibilities [15]. We have recently reported some diorganotin complexes of pyruvic acid isonicotinyl hydrazone and pyruvic acid salicylhydrazone [16]. As an extension of the studies of organotin(IV) complexes with Schiff bases [17-19], we now synthesized seven organotin(IV) complexes with Schiff base of o-vanillin-2-thiophenoylhydrazone. The details of the structure and special characteriza-

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tions of compounds 1–7 are reported herein. Three of the compounds have been studied by X-ray diffraction, and reveal that the compounds have different structure. Furthermore, for compounds 6, 7, because of the presence of big alkyl, the structure of compound 6 presences a monomer and compound 4 rendered a weakly bridged dimer by the weak interactions of Sn...O bond. The Schiff base ligand function as tridentate chelates with O, N and O atoms.

2. Experimental

2.1. Materials and instrumentation

All the solvents used in the reaction were of AR grade and dried using standard literature procedures. The melting points were obtained with Kolfer micro melting point apparatus and were uncorrected. IR spectra were recorded on a Nicolet-460 spectrophotometer using KBr discs. ¹H ¹³C and ¹¹⁹Sn NMR spectra were recorded on a Mercury Plus-400 NMR spectrometer; chemical shifts were given in ppm relative to Me₄Si and Me₄Sn in CDCl₃ solvent. Elemental analyses were performed in a PE-2400 II elemental

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analyzer; the chlorine analyses were performed by the oxygen flask method.

2.2. Preparations of o-vanillin-2-thiophenoylhydrazone

The Schiff base has been prepared with 2-thiophenoylhydrazide and *o*-vanillin in ethanol according to the literature [20]. Yield: 69% (see Scheme 1).

2.3. Preparations of the compounds

Compounds 1-3 were prepared using the same procedure as described for compound 1. The reaction mixture of Schiff base (0.2753 g, 1.0 mmol), triethylamine (1.2 mmol) were added to a solution of ethanol (30 ml), *n*-butyltin trichloride, (0.2822 g, 1.0 mmol) was added, continuing the reaction for 15 h under refluxing. Cooled to room temperature filtered it and gradually removed by evaporation under vacuum until solid product was obtained. The solid was then re-crystallized from dichloromethane-hexane and yellow single crystal was formed by slow evaporation at r.t.

Compounds 4–7 were prepared using the same procedure as described for compound 4. The reaction was carried out under nitrogen atmosphere with use of stranded Schlenk technique. The Schiff base (0.2753 g, 1.0 mmol) was added in the mixture of ethanol and benzene (V/V: 1/3) (30 ml) with sodium ethoxide (0.052 g, 1.0 mmol), the mixture was stirred for 0.5 h, (C₄H₉)₂SnCl₂ (0.3038 g, 1.0 mmol) was added, stirring for 10 h under refluxing. After cooling down to room temperature, filtered it and evaporated to dryness, the solid was then recrystallized from dichloromethane–hexane and yellow single crystal was formed by slow evaporation at r.t.

2.3.1. $[n-C_4H_9SnCl_2(C_{13}H_{11}N_2O_3S)]$ (1)

Yield: 78%, Anal. Calc. for $C_{17}H_{20}Cl_2N_2O_3SSn: C$, 39.11; H, 3.86; N, 5.37; Cl, 13.58. Found: C, 39.03; H, 3.78; N, 5.49; Cl, 13.39%. ¹H NMR (CDCl₃, 400 MHz): 0.95 (t, 3H, -CH₃), 1.34–1.79 (m, 6H, SnCH₂CH₂CH₂--), 3.80 (s, 3H, -OCH₃), 7.17–7.36 (m, 3H, -C₄H₃S), 6.79– 7.12 (m, 3H, Ph--H), 7.80 (s, 1H, C--NH), 8.60 (s, 1H, N=CH). ¹³C NMR (CDCl₃, 400 MHz): 142.55 (CH=N), 168.25 (CO--N), 56.26 (-OCH₃), 149.32, 148.42, 138.12, 131.89, 129.51, 128.25, 122.19, 119.13, 118.92, 114.13 (aromatic carbons), 26.93, 26.65, 20.90, 13.42 (Sn-Bu) ppm. ¹¹⁹Sn NMR (CDCl₃): -343.6 ppm. IR (KBr, cm⁻¹): 3346 (m, NH), 1639 (s, C=N), 1450–1600 (m, Ph), 540 (m, Sn--O), 537 (w, Sn--C).





2.3.2. $[MeSnCl_2(C_{13}H_{11}N_2O_3S)]$ (2)

Yield: 63%. Anal. Calc. for $C_{14}H_{14}Cl_2N_2O_3SSn: C$, 35.03; H, 2.94; N, 5.84; Cl, 14.77. Found: C 34.92; H 2.83; N 5.85; Cl, 14.58%. ¹H NMR (CDCl₃, 400 MHz): 1.22 (s, 6 H, $J_{Sn-H} = 79$ Hz, Sn--CH₃), 3.25 (s, 3H, -OCH₃), 7.15-7.32 (m, 3H, -C₄H₃S), 7.96 (s, 1H, C--NH), 8.51 (s, 1H, N=CH). ¹³C NMR (CDCl₃, 400 MHz): 140.35 (CH=N), 165.68 (CO--N), 56.12 (-OCH₃), 148.78, 147.62, 136.98, 130.49, 128.51, 127.63, 120.37, 117.60, 116.92, 112.05 (aromatic carbons), 15.63 (Sn--CH₃) ppm. ¹¹⁹Sn NMR (CDCl₃): -383.9 ppm. IR (KBr, cm⁻¹): 3323 (m, NH), 1640 (s, C=-N), 1457-1605 (m, Ph), 570 (m, Sn-O), 539 (w, Sn-C).

2.3.3. $[PhSnCl_2(C_{13}H_{11}N_2O_3S)]$ (3)

Yield: 70%. Anal. Calc. for $C_{19}H_{16}Cl_2N_2O_3SSn: C$, 42.10; H, 2.97; N, 5.17, Cl, 13.08. Found: C, 42.03; H, 2.90; N, 5.08; Cl, 12.90%. ¹H NMR (CDCl₃, 400 MHz): 2.49 (s, 3H, --CH₃), 3.38 (s, 3H, --OCH₃), 6.72-7.16 (m, 5H, Ph--H), 7.20-7.38 (m, 3H, --C₄H₃S), 7.84 (s, 1H, C--NH), 8.55 (s, 1H, N=CH). ¹³C NMR (CDCl₃, 400 MHz): 142.35 (CH=N), 173.15 (CO--N), 55.97 (-OCH₃), 150.78, 148.42, 140.12, 135.83, 132.41, 130.56, 125.45, 122.43, 120.37, 117.13 (aromatic carbons), 139.56,139.32, 135.97 136.43, 128.99, 128.76, 126.76 (Sn-Ph) ppm. ¹¹⁹Sn NMR (CDCl₃): -365.6 ppm. IR (KBr, cm⁻¹): 3337 (m, C--NH), 1633 (s, C=N), 1453-1597 (m, Ph), 566 (m, Sn-O), 533 (w, Sn-C).

2.3.4. $[(n-C_4H_9)_2Sn[(C_{13}H_{10}N_2O_3S)]_2(4)$

Yield: 65%, Anal. Calc. for $C_{42}H_{56}N_4O_6S_2Sn_2$: C, 49.72; H, 5.56; N, 5.52; Found: C, 49.55; H, 5.43; N, 5.39%. ¹H NMR (CDCl₃, 400 MHz): 0.91 (t, 12 H, CH₃), 1.32–1.75 (m, 24H, SnCH₂CH₂CH₂), 3.83 (s, 6H, $-OCH_3$), 6.80–6.89 (m, 6H, Ph-H), 7.20–7.38 (m, 6H, $-C_4H_3S$), 8.49 (s, 1H, N=CH). ¹³C NMR (CDCl₃, 400 MHz): 140.36 (CH=N), 164.16 (CO-N), 57.31 ($-OCH_3$), 151.49,150.15, 137.72, 137.45, 135.89, 128.25, 122.97, 121.13, 119.52, 114.13 (aromatic carbons), 30.25, 28.76, 19.86, 15.42 (Sn-Bu) ppm. ¹¹⁹Sn NMR (CDCl₃): -190.6ppm. IR (KBr, cm⁻¹): 1625 (s, C=N-N=C), 1633 (s, C=N), 1459–1610 (m, Ph), 547 (m, Sn-O), 534 (w, Sn-C).

2.3.5. $[(CH_3)_2Sn(C_{13}H_{10}N_2O_3S)]_2$ (5)

Yield: 59%. Anal. Calc. for $C_{30}H_{32}N_4O_6S_2Sn_2$: C, 42.58; H, 3.81; N, 6.62. Found: C, 42.33; H, 3.69; N, 6.50%. ¹H NMR (CDCl₃, 400 MHz): 1.19 (s, 12 H, $J_{Sn-H} = 75$ Hz, Sn-CH₃), 7.23-7.43 (m, 6H, -C₄H₃S), 3.89 (s, 6H, -OCH₃), 6.77-6.86 (m, 6H, Ph-H), 8.53 (s, 2H, N=CH). ¹³C NMR (CDCl₃, 400 MHz): 142.78 (CH=N), 163.89 (CO-N), 55.37 (-OCH₃), 150.98, 150.07, 138.32, 137.65, 136.90, 129.25, 121.68, 121.01, 118.39, 115.36 (aromatic carbons), 16.79 (Sn-CH₃) ppm. ¹¹⁹Sn NMR (CDCl₃): -179.9 ppm. IR (KBr, cm⁻¹): 1616 (s, C=N-N=C), 1639 (s, C=N), 1494-1613 (m, Ph), 563 (m, Sn-O), 539 (w, Sn-C).

2.3.6. $[(C_6H_5)_2Sn(C_{13}H_{10}N_2O_3S)(CH_3OH)]$ (6)

Yield: 73%, Anal. Calc. for $C_{26}H_{24}N_2O_4SSn: C, 53.91$; H, 4.17; N 4.83. Found: C, 53.75; H, 4.06; N, 4.90%. ¹H NMR (CDCl₃, 400 MHz): 3.35 (s, 3H, $-OCH_3$), 3.76 (s, 3H, ArOCH₃), 4.13 (s, 1H, O–H), 6.89–7.06 (m, 3H, Ph–H), 7.19–7.38 (m, 3H, $-C_4H_3S$), 7.45–7.76 (m, 10H, Sn–C₆H₅), 8.60(s, 1H, N=CH). ¹³C NMR (CDCl₃, 400 MHz): 146.73 (CH=N), 170.89 (CO–N), 56.37 ($-OCH_3$), 148.58, 147.27, 138.86, 137.65, 136.09, 129.75, 121.16, 120.36, 118.29, 114.36 (aromatic carbons), 136.29, 130.36, 129.89, 128.95, 126.86, 123.48 (Sn–Ph) ppm. ¹¹⁹Sn NMR (CDCl₃): -168.7 ppm. IR (KBr, cm⁻¹): 3386 v(OH), 1615 (s, C=N–N=C), 1631 (s, C=N), 1453–1600 (m, Ph), 566 (m, Sn–O), 533 (w, Sn–C).

2.3.7. $[(C_6H_5CH_2)_2Sn(C_{13}H_{10}N_2O_3S)]$ (7)

Yield: 69%, Anal. Calc. for $C_{27}H_{24}N_2O_3SSn: C, 56.37$; H, 4.20; N, 4.87. Found: C, 56.20; H, 4.09; N, 4.95%. ¹H NMR(CDCl₃, 400 MHz): 3.25 (t, 4H, $J_{Sn-H} = 88$ Hz, PhCH₂Sn), 3.43 (s, 3H, $-OCH_3$), 6.55–7.63 (m, 16H, Ar–H & $-C_4H_3S$), 8.55 (s, 1H, N=CH). ¹³C NMR (CDCl₃, 400 MHz): 146.78 (CH=N), 169.56 (CO–N), 55.78 ($-OCH_3$), 149.98, 148.07, 138.32, 138.09, 136.46, 129.33, 121.68, 121.01, 118.39, 115.28 (aromatic carbons), 31.63, 139.30, 133.83, 129.36, 128.76, 126.49, 124.56 (Sn–CH₂–Ph) ppm. ¹¹⁹Sn NMR (CDCl₃): -179.5 ppm. IR (KBr, cm⁻¹): 1622 (s, C=N), 1609 (s, C=N–N=C), 1453–1596 (m, Ph), 654 (m, Sn–O), 547 (m, Sn–C).

2.4. Crystallographic measurement

Crystallographic data and refinement details are given in Table 1. All X-ray crystallographic data were collected on a Bruker SMART CCD 1000 diffractometer. A criterion of observability was used for the solution and refinement.

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The structure was solved by a direct method and refined by a full-matrix least-squares procedure based on F^2 using the shelxl-97 program system [21]. All data were collected at 298(2) K using graphite-monochromated Mo K α ($\lambda = 0.71073$ Å) radiation and the ϕ - ω scan technique. All non-hydrogen atoms were included in the model at their calculated positions. The positions of hydrogen atoms were calculated, and their contributions in structure factor calculations were included.

3. Results and discussion of X-ray crystal structures

3.1. Structure of $[n-C_4H_9SnCl_2(C_{13}H_{11}N_2O_3S)]$ (1)

The molecular structure is shown in Fig. 1. The crystallographic data and selected bond lengths and angles are given in Tables 1 and 2. The molecular structure in the Fig. 1 shows that compound 1 is a monomer structure in which the Sn(1) atom and C(1), C(2), C(3), O(1), N(1)atoms form a six membered ring, while the Sn(1) atom and C(9), O(3), N(1), N(2) atoms form a five membered ring. The dihedral angle between the two rings is $3.4(2)^{\circ}$. In compound 1, the tin atom is rendered a distorted octahedral geometry and the equatorially positions were occupied by the nitrogen atom and two oxygen atoms from the tridentate Schiff base ligand, and one C atom from the nbutyl. In the tridentate Schiff base ligand the C-N-N-C chain can indicate by the intermediate C(1)-N(1) [1.285(7) Å], N(1) - N(2) [1.384(6) Å] and N(2) - C(9)[1.340(6) Å]. The axially positions were occupied by two Cl atoms, and the angle of Cl(1)-Sn(1)-Cl(2) is $167.95(5)^{\circ}$, which is deviates from the linear angle 180° . However, the geometry around the tin atom is not as distorted as reported for the other six-coordinated organotin tetradentate ligand compounds [22]. The Sn(1)—O(1) bond

Compound	1	4	6	
Empirical formula	C ₁₇ H ₂₀ Cl ₂ N ₂ O ₃ SSn	$C_{42}H_{56}N_4O_6S_2Sn_2$	C ₂₆ H ₂₄ N ₂ O ₄ SSn	
Formula weight	521.99	8115.26	564.21	
Crystal system	Monoclinic	Monoclinic	Monoclinic	
Space group	$P2_1/C$	$P2_1/C$	$P2_1/C$	
Unit cell dimensions				
a (Å)	9.8759(16)	13.182(4)	11.204(11)	
$b(\mathbf{A})$	18.061(3)	23.643(7)	11.474(11)	
c (Å)	12.328(2)	18.998(4)	19.222(19)	
β (°)	99.897(2)	130.402(13)	99.621(15)	
Volume $(Å^3)$	2166.3(6)	4059(2)	2436(4)	
Z	4	1	4	
Calculated density (mg/m ³)	1.597	1.494	1.538	
<i>F</i> (000)	1036	2064	1136	
Crystal size (mm)	$0.48 \times 0.39 \times 0.23$	$0.44 \times 0.21 \times 0.11$	$0.43 \times 0.39 \times 0.38$	
Scan range θ (°)	2.00-25.01	1.77-25.01	2.07-25.01	
Total/unique/ R_{int}	11161/3819/0.0372	22351/7764/0.0819	12009/4255/0.0733	
Goodness-of-fit on F^2	1.000	1.001	1.002	
Refined parameters	184	505	304	
R_1/wR_2	0.0373/0.0967	0.0835/0.2024	0.0494/0.1148	
$\mu (\mathrm{mm}^{-1})$	1.510	1.249	1.166	
$\rho_{\rm max}/\rho_{\rm min}~({\rm e~A^{-3}})$	0.878 and -0.916	1 1.646 and -0.789	1.323 and -0.832	



Fig. 1. Molecular structure of compound 1.

Table 2											
Selected	bond	distances	(Å) a	ınd	angles	(°)	for	compounds	1.4	and 6	

1			
Sn(1)-O(1)	2.053(3)	N(1) - N(2)	1.384(6)
Sn(1) - N(1)	2.236(4)	N(1)-C(1)	1.285(7)
Sn(1)-O(3)	2.181(3)	N(2)-C(9)	1.340(6)
Cl(1)- $Sn(1)$ - $Cl(2)$	167.95(5)		
4			
Sn(1) - O(1)	2.201(7)	Sn(1)-O(1)#1	2.848(8)
C(18)— $Sn(1)$ — $C(14)$	155.3(5)	O(3) - Sn(1) - N(1)	71.7(4)
C(18)-Sn(1)-O(3)	92.4(5)	C(18) - Sn(1) - O(1)	95.1(4)
C(14)— $Sn(1)$ — $O(3)$	89.6(5)	C(14)— $Sn(1)$ — $O(1)$	94.2(4)
C(18) - Sn(1) - N(1)	100.8(4)	O(3) - Sn(1) - O(1)	153.0(3)
C(14)-Sn(1)-N(1)	103.2(4)	N(1)-Sn(1)-O(1)	81.3(3)
6			
Sn(1) - N(1)	2.152(6)	Sn(1) - O(1)	2.067(5)
C(20)— $Sn(1)$ — $C(14)$	128.9(3)	C(14)— $Sn(1)$ — $O(3)$	94.1(3)
C(20)—Sn(1)—O(3)	93.2(3)		

#1: -x, 2 - y, 1 - z.

length is 2.053(3) Å, and the Sn(1)–O(3) bond length is 2.181(3) Å, which is much less than the sum of the Van der Waals radii for Sn and O (3.68 Å), indicating the significant contacts with the Sn(1) atom. The Sn(1)–N(1) distance is 2.236(4) Å, which is greater than the sum of the non-polar covalent radii 2.15 Å, but is considerably less than the sum of the Van der Waals radii (3.75 Å) [23] and should be considered as bonding interaction, indicating the strong tin–nitrogen interaction.

3.2. Structure of $[(n-C_4H_9)_2Sn[(C_{13}H_{10}N_2O_3S)]_2(4)$ and $[(C_6H_5)_2Sn(C_{13}H_{10}N_2O_3S)] \cdot (CH_3OH)(6)$

The molecular structure of compounds **4** and **6** is shown in Figs. 2 and 3, respectively. The crystallographic data and selected bond lengths and angles are given in Tables 1 and 2. From the Figs. 2 and 3 it can be seen that each tin atom is rendered five-coordinate by coordinating with the nitrogen atom from the Schiff base ligand with a distorted trigonal bipyramidal geometry, surrounded axially by two C atoms from the alkyl and equatorially by one N atom, two O atoms from the Schiff base ligand. So the Schiff base ligand coordinated to the tin atom as a tridentate ligand.

In compound 4 the intramolecular Sn(1)–O(1)bond length of 2.227(10) Å is shorter than those of $\{Ph_2Sn[4-NC_5H_4-C(O)N_2C(CH_3)CO_2](H_2O)\}_2 \cdot CH_2Cl_2 \cdot$ H_2O [2.385(6) Å] and {(PhCH₂)₂Sn[4-NC₅H₄-C(O)N₂C- $(CH_3)CO_2(H_2O)$ [2.383(3) Å] [16], but is longer than the Sn(1)—O(1) bond length of 2.067(5) Å in the compound 6. In compound 4, the Sn(1)-O(1)#1 [#1: -x, 2 - y, 1 - z] distance of 2.848(8) Å is markedly greater than the sum of the covalent radii for Sn-O, but is considerably less than the sum of the Van der Waals radii. It is shown that the O(1)#1 atom makes weak contacts with the Sn(1) atom. Two molecules form a weak-bridged dimer with weak interactions of Sn...O bonding. While in compound 6, there was no intramolecular interaction between the Sn(1)atom and O(1)#1 atom, which is due to the presence of the big phenyl and make the compound stay as a monomer. The packing of the molecules in a unit cell of compound 6 can confirm this. The Sn(1)-N(1) bond length are 2.227(10) and 2.152(6) Å in compounds 4 and 6, which is shorter than the bond length in compound 1, indicating the strong tin-nitrogen interaction.

The distorted trigonal bipyramidal geometry around the tin atom is a result of the strain imposed by the tridentate Schiff base ligand, and from the constraints imposed by the five and six membered ring Sn-N-N-C-O and Sn-N-C-C-C-O. This is reflected in compound 4, the angles of C(18)-Sn(1)-O(3) 92.4(5)°, C(18)-Sn(1)-O(1) 95.1(4)° and C(14)-Sn(1)-O(1) 94.2(4)° are greater than



Fig. 2. Molecular structure of compound 4.



Fig. 3. Molecular structure of compound 6.

90°. In contrast, the angles of C(14)–Sn(1)–O(3) 89.6(5)°, O(3)–Sn(1)–N(1) 71.7(4)° and O(1)–Sn(1)–N(1) 81.3(3)° are less than 90°. The distorted geometry can also be seen in the deviation from 180° of the angle C(18)–Sn(1)–C(14) 155.3(5)°. While in compound **6** the angle of C(20)– Sn(1)–C(14), C(20)–Sn(1)–O(3) and C(14)–Sn(1)–O(3) are 128.9(3)°, 93.2(3)° and 94.1(3)° can also indicate the distorted trigonal bipyramidal geometry. Those conclusions are supported by the X-ray single crystal diffraction study.

4. Supplementary material

Crystallographic data for compounds 1, 4, 6 have been deposited at the Cambridge Crystallographic Data Center as supplementary publication numbers CCDC 293293, 291922 and 292084, respectively. Copies of available material may be obtained on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336033; or e-mail: deposit@ccdc.cam.ac.uk).

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