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Syntheses, Characterizations and Crystal Structures of New Triorganotin Complexes with 2-Mercaptopyrimidine and 4-Amino-2-Mercaptopyrimidine

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ABSTRACT: *Four triorganotin(IV) complexes with 2-mercaptopyrimidine (HSpym) and 4-amino-2-mercaptopyrimidine (HSapym) of the type, R_3SnL (L= Spym, R=Ph, 1; R=$PhCH_2$, 2; L=Sapym, R=Ph, 3; R=$PhCH_2$, 4), were synthesized. All the complexes* **1–4** *have been characterized by elemental, IR, ^1H NMR, and X-ray crystallography diffraction analyses, which revealed that the structures of* **1–4** *are penta-coordinated with R_3Sn-coordinated to the thiol S and heterocyclic N atoms, and the structural distortion for each is a displacement from tetragonal toward trigonal bipyramidal geometry. The complex* **1** *is a one-dimensional chain complex, while compounds* **3** *and* **4** *are dimers due to the existence of N···H hydrogen bonding.* © 2005 Wiley Periodicals, Inc. Heteroatom Chem 16:69–75, 2005; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20068

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INTRODUCTION

Since some organotin complexes were found to exhibit the properties of antitumor activity in vitro, many series of organotin(IV) complexes have been synthesized and studied in the context of their antitumor potential [1]. Among these efforts to obtain potential antitumor organotin(IV) species, an important strategy is to synthesize organotin(IV) derivatives of biologically relevant substrates. Many investigations involved nucleic acid derivatives [2–4], amino acids derivatives, and so on [5–8]. It is proved that the 2-mercaptopyrimidine (HSpym) and 2-mercapto-4-aminopyrimidine (HSapym), similar to 2-mercaptopyrimidine nucleotides, are able to inhibit the synthesis of tRNA [9]. Thus they may act as valuable substrates for synthesizing antitumor-active organotin compounds. Moreover, as far as the coordination chemistry is concerned, the two ligands are interesting too. Both of them own one deprotonated heterocyclic thioamide group $(N\text{-}C\text{-}S)^-$ and can act as *S, N*-bridging ligands. Especially, for Hsapym, the amino group may offer the second interaction potential.

Out of above consideration, also as a part of our studies on coordination chemistry of heterocyclic thiones with organotin(IV) complexes [10–13], we report here on the syntheses and characterizations of four trialkyltin complexes with 2-mercaptopyrimidine (HSpym) and 4-amino-2-mercaptopyrimidine (HSapym) of the type R_3SnL

(L=Spym, R=Ph, **1**; R=$PhCH_2$, **2**; L=Sapym, R=Ph, **3**; R=$PhCH_2$, **4**). All the complexes **1–4** have been characterized by elemental, IR, ^{1}H NMR, and X-ray crystallography diffraction analyses, which revealed that the structures of **1–4** are penta-coordinated with R_3Sn-coordinated to the thiol S and heterocyclic N atoms, and the structural distortion for each is a displacement from tetragonal toward trigonal bipyramidal geometry. The complex **1** is a one-dimensional (1D) chain complex, while compounds **3** and **4** are two dimers due to the existence of N···H hydrogen bonding.

EXPERIMENTAL

Materials and Measurements

All reagents and solvents were purchased commercially and used without further purification unless otherwise noted. The melting points were obtained on a Kofler micromelting point apparatus and are uncorrected. Infrared spectra were recorded on a Nicolet-460 spectrophotometer using KBr disks and sodium chloride optics.^{1}H NMR spectra were obtained on a Jeol-FX-90Q NMR spectrometer. Chemical shifts are given in ppm relative to Me_4Si in $CDCl_3$ as solvent. Elemental analyses were performed with a PE-2400 II elemental analyzer.

Syntheses of the Complexes **1–4**

*[Ph_3Sn(Spym)] (**1**).* Under nitrogenous atmosphere the 2-mercaptopyrimidine (0.112 g, 1 mmol), sodium ethoxide (0.068 g, 1 mmol), and triphenyltin chloride (0.385 g, 1 mmol) were added in turn in benzene (20 mL), stirred for 12 h at 40°C and filtrated and then obtained the solid, recrystallized the solid in diethylether-petroleumether and complex **1** was formed. Yield: 81%. Mp 118°C. Anal. found: C, 57.41; H, 3.83; N, 6.14; calcd. for $C_{22}H_{18}N_2SSn$: C, 57.30; H, 3.93; N, 6.08%. 1H NMR ($CDCl_3$, δ ppm): 7.38–7.74 (m,18H,aromatic-H). IR(KBr): 281 ν(Sn—S); 562 ν_{as}(Sn—C); 544 ν_s(Sn—C); 446 ν(Sn—N); 730 ν(C—S); 1563ν (C=N).

*[$(PhCH_2)_3$ Sn(Spym)] (**2**).* The procedure is similar to that of complex **1**, and crystalline complex **2** was formed in diethyl ether–petroleum ether. Yield: 86%. Mp 98–100°C. Anal. found: C,59.58; H, 4.72; N, 5.68; calcd. for $C_{25}H_{24}N_2SSn$: C,59.67; H,4.81; N, 5.57%. 1H NMR ($CDCl_3$, δ ppm): 6.77–7.26 (m, 18H, aromatic-H), 2.58 (s, 6H). IR(KBr): 292 ν(Sn—S); 570 ν_{as}(Sn—C); 549 ν_s (Sn—C); 448 ν(Sn—N); 1566 ν(C=N).

*[Ph3Sn(Sapym)] (**3**).* The procedure is the same as that of complex **1**, and crystalline complex **3** was formed in diethyl ether–petroleum ether. Yield: 83%. Mp 166–168°C. Anal. found: C, 55.63; H, 4.13; N, 8.69; calcd. for $C_{22}H_{19}N_3SSn$: C, 55.50; H, 4.02; N, 8.82%. 1H NMR ($CDCl_3\delta$ ppm): 7.26–7.78(m, 17H, aromatic-H), 6.02 (s, 2H). IR (KBr): 286 ν(Sn—S); 566 ν_{as}(Sn—C); 476 ν_s(Sn—C); 443 ν(Sn—N); 732 ν(C—S); 1580 ν(C=N).

*[$(PhCH_2)_3$ Sn(Sapym)] (**4**).* Crystalline complex **4** was made in the same way of complex **1**. It was formed in ether–hexane. Yield: 84%. Mp 128–130°C. Anal. found: C, 57.83; H, 4.93; N, 8.24; calcd. for $C_{25}H_{25}N_3SSn$: C, 57.94; H, 4.86; N, 8.11%. 1H NMR ($CDCl_3$ δ ppm): 6.80–7.64 (m, 17H), 6.02 (d, 2H), 2.54 (s, 6H). IR (KBr): 297 ν(Sn—S); 478 ν_{as}(Sn—C); 423 ν_s(Sn—C); 451 ν(Sn—N); 1580 ν(C=N).

X-Ray Crystallography

All X-ray crystallographic data were collected on a Bruker SMART CCD 1000 diffractometer. Correction for semi-empirical from equivalents was applied, and the structure was solved by direct methods and refined by a full-matrix least squares procedure based on F^2 using the SHELXL-97 program system. All data were collected with graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å) at 298(2) K. The positions of hydrogen atoms were calculated, and their contributions were included in structural factor calculations.

RESULTS AND DISCUSSION

Syntheses of the Complexes **1–4**

The synthesis procedure was as in the following Scheme 1:

$$R_3SnCl + HL + EtONa \longrightarrow R_3SnL$$

(L=Spym, R=Ph , **1**; R=$PhCH_2$, **2** ; L=Sapym, R=Ph, **3**; R=$PhCH_2$, **4**)

SCHEME 1

IR Spectroscopic Studies of the Complexes **1–4**

The explicit feature in the infrared spectra of all complexes **1–4** is the absence of the band in the region 2514 cm^{-1}, which appears in the free-ligand as the ν(S—H) vibration. While in the far-infrared spectra, strong absorption appears at the range of 283–306 cm^{-1} in the respective spectra of the complexes **1–4**, which is absent in the spectrum of the ligand,

and is assigned to the Sn—S stretching mode of vibration. All these values are consistent with that detected in a number of organotin(IV)-sulfur derivatives and indicate the formation of metal—ligand bond through this site [14].

The ν(C=N) band, occurring at about 1564–1580 cm^{-1}, is considerably shifted toward lower frequencies with respect to that of the free ligand (1608 cm^{-1}), confirming the coordination of the heterocyclic N atom to tin. The stretching frequency is lowered because of the displacement of electron density from N to the Sn atom, thus resulting in a weakening of the C=N bond as reported in the literature [15].

Besides, in organotin complexes, the IR spectra can provide useful information concerning the geometry of the SnC_n moiety [16]. In the case of our complexes, the bands at 562 and 544 cm^{-1} for **1**, at 570 and 549 cm^{-1} for **2**, 566 and 476 cm^{-1} for **3**, and 570 and 544 cm^{-1} for **4** can be assigned to ν_{as}(Sn—C) and ν_s(Sn—C), respectively, suggesting noplanar SnC_3 fragments for trialkyltin derivatives **1–4**.

^{1}*H NMR Data of the Complexes* **1–4**

^{1}H NMR data showed that the signal of the —SH proton in the spectrum of the ligand is absent in all the complexes **1–4**, indicating the removal of the —SH proton. This information agrees with what the IR data have revealed.

Crystal Structures of Complexes **1–4**

The crystal structures or unit cells of complexes **1–4** in crystal are shown in Figs. 1, 2, 3, and 4 respectively. All H atoms have been omitted for the purpose of clarity. Table 1 lists the crystal data and structure refinement parameters for complexes **1–4**, and their selected bond lengths and angles are shown in Tables 2 and 3, respectively.

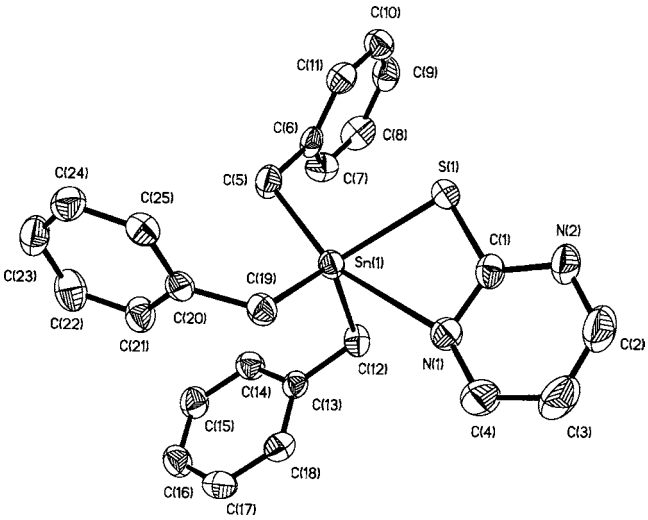

FIGURE 2 Crystal structure of complex **2**.

Structure of Complex 1 and 2. Both of the tin atoms in complexes **1** and **2** are five coordinated. The ligand HSpym acts as a bidentate *S, N*-chelating ligand. The X-ray structure of complex **1** has been earlier reported by Pettrilli et al. [17]. For the benefit of clarifying the discussion of the spectroscopic results (see above) and take a useful comparison, we have studied this compound for a second time and described the X-ray structure here. As shown in Fig. 1, the central tin atom of complex **1** forms four primary bonds: three to the phenyl

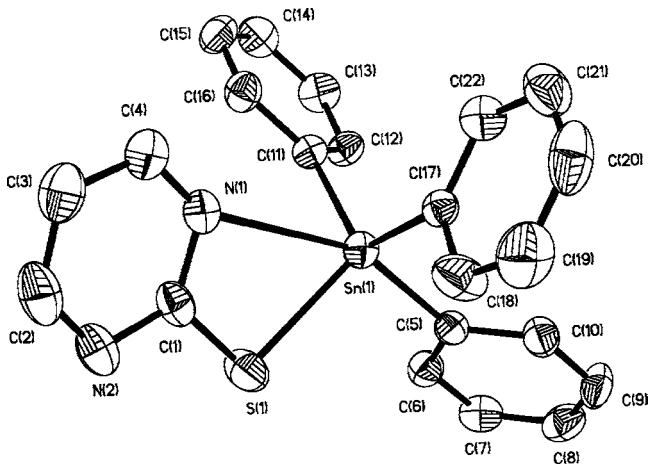

FIGURE 1 Crystal structure of complex **1**.

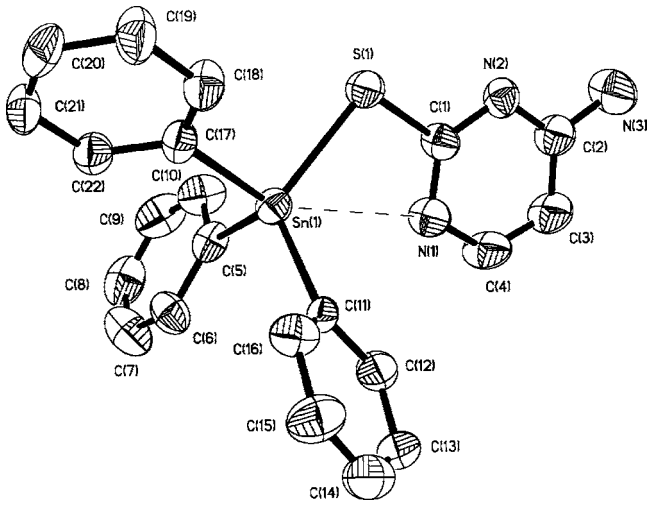

FIGURE 3 Crystal structure of complex **3**.

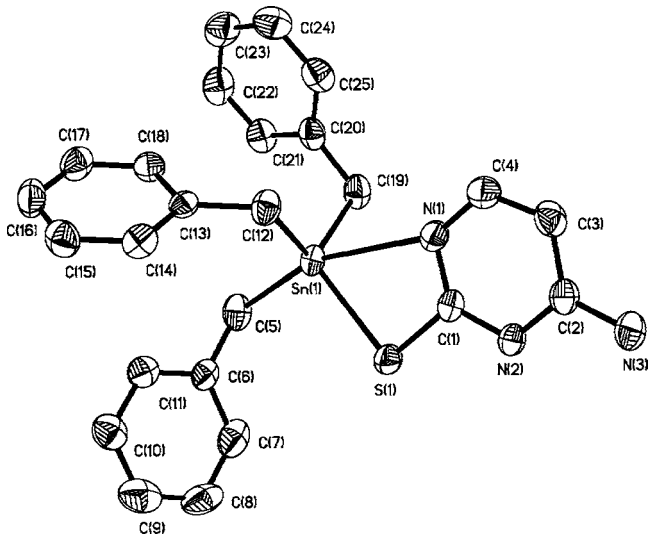

FIGURE 4 Crystal structure of complex **4**.

groups and one to the sulfur atom. In addition, there is a weak intramolecular Sn—N interaction, the Sn(1)—N(1) bond length (2.878 Å) is longer than the sum of covalent radii (2.15 Å), but is considerably shorter than that reported in $Ph_3Sn(MBZ)$ (3.07 Å) [14]. Thus complex **1** has a distorted *cis*-trigonal-bipyramidal geometry with the sulfur (S(1)) atom and two carbon atoms (C(11) and C(17)) occupying the equatorial plane, whereas the nitrogen (N(1)) and another carbon atom (C(5)) are in axial positions. The sum of the equatorial angles around the tin atom, (C(11)–Sn(1)–S(1), (17)–Sn(1)–S(1), and C(11)–Sn(1)–C(17)), is 342.09° and the axial-Sn-axial angle, C(5)–Sn(1)–N(1), is 156.79(14)°. The significant distortion is mainly due to the rigidity of the chelate ring (N(1)–Sn(1)–S(1): 58.42°), together with the large covalent radius of tin(IV). The Sn(1)–S(1) bond length (2.440(15) Å) lies in the range reported of those penta-coordinated organotin complexes (2.41–2.48 Å) [18–20].

It is worth noting that there exists intermolecular N···H—C hydrogen bonding between nitrogen (N(2)) and phenyl hydrogen atoms. The N···H—C distance is 2.690 Å for C(20)–H(20)···N(2)#, which approaches to the van der Waals radii of the two atoms (2.70 Å), which indicates that the interaction is very weak. And by the linkage of the N···H—C hydrogen bonding, complex **1** exists as a 1D chain complex.

As shown in Fig. 2, complex **2** has a distorted *cis*-trigonal-bipyramidal geometry with a tin–nitrogen

TABLE 1 Crystal Data and Structure Refinement Parameters for Complexes **1, 2, 3**, and **4**

	Complex **1**	*Complex* **2**	*Complex* **3**	*Complex* **4**
Empirical formula	$C_{22}H_{18}N_2SSn$	$C_{25}H_{24}N_2SSn$	$C_{22}H_{19}N_3SSn$	$C_{25}H_{25}N_3SSn$
Formula weight	461.13	503.21	476.15	518.23
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	*P*2(1)/*c*	*P*2(1)/*c*	*P*2(1)/*n*	*P*2(1)/*n*
Unit cell dimensions				
a (Å)	8.701(4)	10.311(3)	16.252(9)	12.19(2)
b (Å)	35.892(17)	19.261(7)	9.451(5)	12.98(3)
c (Å)	6.589(3)	11.620(4)	16.530(9)	15.38(3)
α (°)	90	90	90	90
β (°)	100.045(8)	92.082(4)	111.752(8)	103.64(3)
γ (°)	90	90	90	90
V (Å3)	2026.3(17)	2306.1(13)	2358(2)	2365(8)
Z	4	4	4	4
D_c (g cm^{-3})	1.512	1.449	1.341	1.456
Crystal size (mm)	0.42 × 0.35 × 0.09	0.33 × 0.25 × 0.20	0.35 × 0.27 × 0.15	0.42 × 0.37 × 0.25
F(000)	920	1016	952	1048
Total reflections	10591	11979	8797	9551
Independent reflections	3559	4070	3797	4122
Data/restraints/parameters	3559/0/235	4070/0/262	3797/0/244	4122/0/271
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0401$	$R_1 = 0.0264$	$R_1 = 0.0761$	$R_1 = 0.0290$
	$wR_2 = 0.0577$	$wR_2 = 0.0497$	$wR_2 = 0.1725$	$wR_2 = 0.0648$
R indices (all data)	$R_1 = 0.0745$	$R_1 = 0.0435$	$R_1 = 0.1335$	$R_1 = 0.0473$
	$wR_2 = 0.0634$	$wR_2 = 0.0534$	$wR_2 = 0.2097$	$wR_2 = 0.0728$
Goodness-of-fit	0.909	0.894	0.944	1.030
Theta range for data collection (°)	2.27–25.03	1.98–25.03	2.63–25.02	1.93–25.02
Absorption coefficient (mm^{-1})	1.371	1.211	1.182	1.185
Largest diff. Peak and hole(e. Å^{-3})	0.432 and −0.327	0.433 and −0.308	1.061 and −0.733	0.390 and −0.541

TABLE 2 Selected Bond Lengths (Å) and Angles (°) of Complexes **1** and **3**

Complex **1**		*Complex* **3**	
Bond lengths			
Sn(1)–C(11)	2.126(4)	Sn(1)–C(11)	2.150(6)
Sn(1)–N(1)	2.878(4)	Sn(1)–C(5)	2.151(7)
Sn(1)–S(1)	2.4400(15)	Sn(1)–C(17)	2.180(8)
Sn(1)–C(5)	2.141(4)	Sn(1)–S(1)	2.447(2)
Sn(1)–C(17)	2.127(4)	Sn(1)–N(1)	3.009(7)
S(1)–C(1)	1.753(5)	S(1)–C(1)	1.767(8)
N(1)–C(1)	1.319(5)	N(1)–C(1)	1.315(10)
N(2)···H(20)–C(20)	2.690	N(2)···H(3)–N(3)	2.302
Bond angles			
C(5)–Sn(1)–N(1)	156.79(14)	C(11)–Sn(1)–C(5)	111.5(3)
C(17)–Sn(1)–S(1)	109.25(13)	C(11)–Sn(1)–C(17)	106.6(3)
C(11)–Sn(1)–N(1)	88.40(14)	C(5)–Sn(1)–C(17)	106.9(3)
C(5)–Sn(1)–S(1)	99.29(12)	C(11)–Sn(1)–S(1)	118.55(19)
C(17)–Sn(1)–N(1)	79.29(13)	C(5)–Sn(1)–S(1)	114.1(2)
C(11)–Sn(1)–S(1)	113.52(12)	C(17)–Sn(1)–S(1)	97.2(2)
C(11)–Sn(1)–C(17)	119.32(17)	C(11)–Sn(1)–N(1)	83.6(2)
C(17)–Sn(1)–C(5)	104.97(17)	C(5)–Sn(1)–N(1)	91.6(3)
S(1)–Sn(1)–N(1)	58.42(9)	C(17)–Sn(1)–N(1)	153.0(2)
C(11)–Sn(1)–C(5)	108.20(17)	S(1)–Sn(1)–N(1)	56.69(14)

intramolecular interaction similar to complex **1**. The chelate bite angle, N(1)–Sn(1)–S(1), is 60.65(6)°. The axial-Sn-axial angle (C(5)–Sn(1)–N(1), 160.17(10)°), strongly affected by the small bite angle, is well removed from the ideal trigonal-bipyramidal angle. The Sn—S bond length is 2.46 Å, which lies in the normal range for those higher than four-coordinated organotin structures but is shorter than that reported in $(PhCH_2)_3Sn(MNBT)$(2.49 Å) [21]. Concerning the Sn—N bond, the bond length (Sn(1)–N(1): 2.724(2) Å) is just between the sum of the van der Waals and covalent radii of Sn and N (3.75 and 2.15 Å, respectively) [22] but shorter than that in complex **1** (2.878(4) Å), which evidences the influence of stereo-constraints, the larger the stereo-constraint of R groups, the more difficult the substituent for nitrogen atom. In this situation, the stereo-constraint of phenyl group is larger than that of benzyl group, so that the Sn—N bond of complex **2** is stronger than that of complex **1**.

TABLE 3 Selected Bond Lengths (Å) and Angles (°) of Complexes **2** and **4**

Complex **2**		*Complex* **4**	
Bond lengths			
Sn(1)–C(5)	2.175(3)	Sn(1)–C(12)	2.143(4)
Sn(1)–C(12)	2.164(3)	Sn(1)–C(19)	2.150(5)
Sn(1)–C(19)	2.155(3)	Sn(1)–C(5)	2.166(5)
Sn(1)–S(1)	2.4559(10)	Sn(1)–S(1)	2.455(4)
Sn(1)–N(1)	2.724(2)	Sn(1)–N(1)	2.777(6)
S(1)–C(1)	1.752(3)	S(1)–C(1)	1.759(5)
N(1)–C(1)	1.336(4)	N(1)–C(1)	1.319(5)
Bond angles			
C(19)–Sn(1)–S(1)	119.13(8)	C(12)–Sn(1)–C(19)	112.45(19)
C(19)–Sn(1)–C(12)	113.78(12)	C(12)–Sn(1)–C(5)	111.63(14)
N(1)–Sn(1)–S(1)	60.65(6)	C(19)–Sn(1)–C(5)	104.78(3)
C(5)–Sn(1)–C(12)	107.90(12)	C(12)–Sn(1)–S(1)	110.75(15)
C(19)–Sn(1)–N(1)	82.07(9)	C(19)–Sn(1)–S(1)	115.70(10)
S(1)–Sn(1)–C(12)	108.08(8)	C(5)–Sn(1)–S(1)	100.74(12)
C(5)–Sn(1)–N(1)	160.17(10)	C(12)–Sn(1)–N(1)	77.78(12)
C(19)–Sn(1)–C(5)	106.62(11)	C(19)–Sn(1)–N(1)	86.03(13)
S(1)–Sn(1)–C(5)	99.91(8)	C(5)–Sn(1)–N(1)	160.51(13)
C(12)–Sn(1)–N(1)	83.66(9)	S(1)–Sn(1)–N(1)	59.78(7)

Moreover, it is worthwhile to note that both the Sn—N bond lengths of complexes **1** and **2** are consistent with the average Sn—N bond length (>2.39 Å) of active Sn complexes having antitumor activity [23].

Structures of Complex 3 and 4. As shown in Figs. 3 and 4, both of the tin atoms in complexes **3** and **4** are five coordinated. And the ligand Hsapym acts as a *S,N*-chelating ligand. The amino group does not involve any interaction with tin atoms but its existence does influence the coordination priority of the heterocyclic nitrogen atoms to tin atom. In the cases of complexes **3** and **4**, it is N(1) but not N(2) that involves the coordination to tin atoms.

For complex **3**, tin coordinates to three carbon atoms and a sulfur atom of Sapym group. The trigonal equatorial plane is occupied by the sulfur (S(1)) atom and two carbon atoms (C(11) and C(5)), whereas the axial positions was located by nitrogen (N(1)) and another carbon atom (C(17)) with the axial-Sn-axial angle(C(17)–Sn(1)–N(1)), 153.0(2)°. The sum of the equatorial angles around the tin atom, (C(11)–Sn(1)–S(1), C(5)–Sn(1)–S(1), and C(11)–Sn(1)–C(5)) is 344.15°. The Sn(1)–S(1) bond length (2.447 (2) Å) is almost equal to that of complexes **1** and is longer than that in $Ph_3Sn(Me_2Pymt)$ (2.433 Å) [24], within the range (2.405–2.481 Å) for triphenyltin(IV) thiolates compounds R_3SnL reported before [25,26]. The Sn(1)–N(1) bond length (3.009 Å) is shorter than those in $Ph_3Sn(MBZ)$ (3.07 Å) [14] and $Ph_3Sn(MNBT)$ (3.14 Å) [27] but longer than that in complex **1** (2.878 Å) and complex $Ph_3Sn(Me_2Pymt)$ (2.835 Å) [24].

It should be noted that intermolecular N···H hydrogen bonding is recognized between nitrogen (N(2)) and amino hydrogen atoms. The average N···H distance is 2.302 Å, which is shorter than those in complex **1**. And it is the very intermolecular hydrogen bonding that made complex **3** a dimer.

For complex **4**, similar to complexes **2** and **3**, the geometry of tin atom is also distorted *cis*-trigonal-bipyramidal. N(1)–Sn(1)–S(1) is 59.78(7)°. C(5)–Sn(1)–N(1) is 160.51(13)°. The Sn—S bond length (2.455 Å) is consistent with that of complex **2**. Concerning the Sn—N bond, the bond lengths (Sn(1)–N(1): 2.777 Å) is slightly longer than that in complex **2** (2.724 Å) but much shorter than that in complex **3** (3.009 Å), revealing the weaker stereo-constraint of $PhCH_2$-compared with that of Ph—.

Furthermore the complex **4** is made a dimer by N···H hydrogen bonding existing between nitrogen (N(2)) and amino hydrogen atoms. The N···H distance (2.185 Å) is much shorter than the van der Waals radii of the two atoms (2.700 Å) and also shorter than complex **3**.

CONCLUSIONS

A series of organotin complexes based on two *S,N*-chelating ligands: 2-mercaptopyrimidine and 4-amino-2-mercaptopyrimidine have been synthesized and characterized. Detailed studies on the structures and spectra of these complexes indicate that their structures are all distorted trigonal bipyramidal geometry. Besides, the stereo-constraints of R groups of R_3SnCl were found to have great influence to the Sn—N bond intensity, the trend is that those Sn—N bond in Ph_3SnL is weaker than those in $(PhCH_2)_3SnL$. Specially, for $R_3Sn(Sapym)$, the stereo-constraint of amino group, together with the R groups of R_3SnCl, makes the coordination trend of N(1) to tin atom larger than that of N(2). Furthermore N···H hydrogen bonding was recognized in most of these complexes, which helps to construct their 1D chain or dimer structure.

SUPPLEMENTARY MATERIAL

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC nos. 233177, 235028, 227962, and 227960 for complexes **1, 2, 3**, and **4** respectively. Copies of these information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB21EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam. ac.uk or www: http://www.ccdc.cam.ac.uk).

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