

Communication

Solvothermal synthesis and crystal structure of a mixed tri- and dimethyltin coordination polymer, $\{[(\text{Me}_3\text{Sn})(\mu_2\text{-OH})][\text{Me}_2\text{Sn}(\mu_3\text{-SCH}_2\text{CO}_2)]\}_n$

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Abstract

A mixed tri- and dimethyltin coordination polymer, $\{[(\text{Me}_3\text{Sn})(\mu_2\text{-OH})][\text{Me}_2\text{Sn}(\mu_3\text{-SCH}_2\text{CO}_2)]\}$ (**1**), has been solvothermally synthesized and structurally characterized by elemental analysis, FT-IR, NMR (¹H, ¹³C, ¹¹⁹Sn) spectra and X-ray crystallography. This complex displays a 1D helical chain structure, in which it can be considered as the tri- and dimethyltin moieties are linked together by the alternate $\mu_2\text{-OH}$ groups and $[\mu_3\text{-SCH}_2\text{CO}_2]^{2-}$ units along the $[10\bar{1}]$ direction. Furthermore, these helical chains are linked together through inter-helix O–H···S H-bonding interactions to form a 2D corrugated sheet.

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1. Introduction

Recently, organotin complexes have been intensely studied owing to their potential biological activities as well as their wide industrial and agricultural applications [1,2]. Among the organotin complexes, organotin carboxylates are one of the most ubiquitous for their considerable structural diversity, such as monomers, dimers, tetramers, oligomers and polymers, etc [3]. It has also been demonstrated that other structural types are formed due to the presence of additional coordinating sites (e.g., N, O or S) along with a carboxylate moiety [4–6]. Although considerable advance has been reached in the development of organotin chemistry, mixed tri- and diorganotin carboxylates have been rarely reported so far [7]. Basu Baul and his co-workers have reported several monomeric mixed tri- and diorganotin complexes derived from Schiff base ligands, where the

tin atoms in diorganotin moieties have been involved in intramolecular Sn ← N interactions [7]. More recently, polymeric organotin carboxylates have attracted our more attention due to their interesting and various supramolecular architectures and topologies [8,9]. In our previous work, we have reported several novel and interesting organotin polymers with carboxylate ligands [9]. To continue our research in this area, we select another interesting carboxylate ligand: mercaptoacetic acid. The ligand was chosen by reason that the thiol group in this ligand can assist the carboxyl group to form polymeric structure, and also that the organotin sulfides have good thermal stability and can be used as stabilizers for PVC and related materials [10]. As literatures reported, some organotin derivatives with mercaptoacetic acid have already been synthesized and characterized under mild conditions [11]. Therefore, our interest has been focused on investigating the reaction of these simple organotin compounds and mercaptoacetic acid under extreme condition and hoping to obtain novel organotin complexes that can not get them under the mild condition.

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On the other hand, solvothermal synthesis has become an efficient method for preparing variously novel coordination complexes using the simple inorganic or organometallic compounds and organic ligands as starting materials, and it is also a superb technique for crystallizing these products that hardly arise from other synthetic methods [12]. We have previously obtained a 12-membered cyclic organotin complex using the solvothermal technique [13]. And now we are interested in exploring the reaction of trimethyltin chloride and mercaptoacetic acid under solvothermal condition and reported herein the crystal structure of the resulting product—a mixed tri- and dimethyltin coordination polymer, $\{[(\text{Me}_3\text{Sn})(\mu_2\text{-OH})][\text{Me}_2\text{Sn}(\mu_3\text{-SCH}_2\text{CO}_2)]\}_n$ (**1**).

2. Results and discussion

2.1. Synthesis and spectra

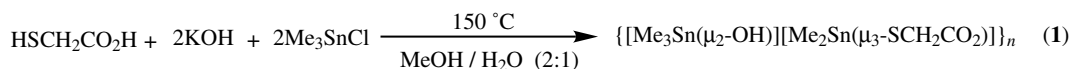
Mercaptoacetic acid, trimethyltin chloride, and KOH have been reacted with 1:2:2 molar ratio in the mixed solution of CH_3OH and H_2O ($v/v = 2:1$) under solvothermal condition ($150\text{ }^\circ\text{C}$) and yielded the mixed tri- and dimethyltin polymer, $\{[(\text{Me}_3\text{Sn})(\mu_2\text{-OH})][\text{Me}_2\text{Sn}(\mu_3\text{-SCH}_2\text{CO}_2)]\}_n$ (**1**) (Scheme 1). It can be dissolved in common polar solvent, such as chloroform, ethanol, methanol, acetonitrile and dimethylsulfoxide, etc.

In the IR spectrum of complex **1**, a broad absorption at 3050 cm^{-1} is attributed to $\nu(\text{O-H})$ stretching vibration, and the corresponding Sn–O (461 cm^{-1}) and Sn–S (325 cm^{-1}) absorption values are all located in the range of that has

been reported in the similar organotin complexes [14]. The $\Delta\nu$ (202 cm^{-1}) values [$\Delta\nu = \nu(\text{CO}_2)_{\text{as}} - \nu(\text{CO}_2)_{\text{s}}$] indicate the $-\text{CO}_2$ group are coordinated with the bidentate mode in complex **1** [15]. The ^1H NMR data and ^{13}C NMR spectral pattern are consistent with the formulation of complex **1**. The ^{119}Sn NMR spectrum of complex **1** exhibits two signals at -128.6 and -195.5 ppm, suggesting both the two tin atoms are five-coordinated and their coordination environment are not completely identical in the solution [7,16].

2.2. Description of crystal structure

The crystal structure of **1** is constructed from a 1D helical chain consisting of $\{[(\text{Me}_3\text{Sn})(\mu_2\text{-OH})][\text{Me}_2\text{Sn}(\mu_3\text{-SCH}_2\text{CO}_2)]\}_n$ entities. Fig. 1 shows a perspective view of the $\{[(\text{Me}_3\text{Sn})(\mu_2\text{-OH})][\text{Me}_2\text{Sn}(\mu_3\text{-SCH}_2\text{CO}_2)]\}$ entity with the atomic numbering scheme, in which the oxygen atom of O2A comes from another carboxylate so as to achieve a five-coordinate geometry of the Sn2 atom. The asymmetric unit, $\{[(\text{Me}_3\text{Sn})(\mu_2\text{-OH})][\text{Me}_2\text{Sn}(\mu_3\text{-SCH}_2\text{CO}_2)]\}$, consists of a trimethyltin hydroxide moiety, $[(\text{Me}_3\text{Sn})(\mu_2\text{-OH})]$, and a cyclic dimethyltin moiety, $[\text{Me}_2\text{Sn}(\mu_3\text{-SCH}_2\text{CO}_2)]$. The trimethyltin hydroxide moiety can be considered as a hydrolyzed product derived from Me_3SnCl , which has been reported many years ago by Kasai et al. [17]. And the dimethyltin moiety has a five-membered SnSCCO chelate metalocycle with the envelope conformation [11a]. Interestingly, the five atoms in the chelate ring (Sn1, S1, C1, C2 and O1) and the two O atoms (O2 and O3) in each unit almost lie in the same plane (largest deviation: 0.097 \AA),



Scheme 1.

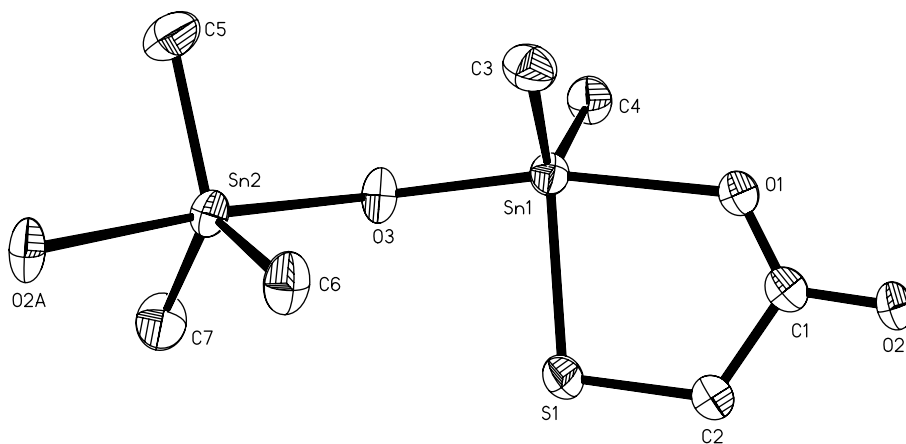


Fig. 1. A monomer fragment of the 1D helical chain with the atomic labeling scheme in complex **1**. Selected bond distances (\AA) and bond angles ($^\circ$): Sn1–O1 2.268(4), Sn1–O3 2.118(4), Sn1–S1 2.4230(16), Sn2–O3 2.243(4), Sn2–O2A 2.303(4), C1–O1 1.255(7), C1–O2 1.240(6); C3–Sn1–C4 118.7(2), C3–Sn1–S1 120.43(17), C4–Sn1–S1 119.5(2), O1–Sn1–O3 167.35(16), C5–Sn2–C6 119.4(3), C6–Sn2–C7, 120.6(3), C5–Sn2–C7 119.6(3), O2A–Sn2–O3 173.29(14). (symmetry code: A $x + 1/2, -y + 1/2, z - 1/2$).

but the Sn2 displaced a large distance (1.633 Å) out of this plane on account of the hydroxyl O atom (O3) hybridized with aniso- sp^3 type. Both tin atoms in complex **1** are five-coordinated, but the coordinate environments of them are not completely identical. For the tin atom in the trimethyltin moiety, Sn2, it is coordinated by five atoms with the three methyl groups forming the equatorial plane and the hydroxyl O atom (O3) and one of the carboxyl O atom (O2A, symmetry code: $x + 1/2, -y + 1/2, z - 1/2$) at axial positions (O3–Sn2–O2A, 173.31°). For the tin atom in dimethyltin moiety, Sn1, it is also coordinated by five atoms but with the two methyl groups and the thiol S atom (S1) forming the equatorial plane and the hydroxyl O atom (O3) and the other carboxyl O atom (O1) at axial positions (O3–Sn1–O1, 167.36°). There is a strong {S, O} chelate effect in the five-membered chelate ring (S1–Sn1–O1 = 79.64°), so the axial–Sn1–axial angle (167.36°) is smaller than the axial–Sn2–axial (173.31°) and the distance of Sn1 atom deviated from the defined equatorial plane (0.114 Å) is also larger than that found in Sn2 atom (0.055 Å). The two Sn–O bond lengths around Sn2 atom (Sn2–O2A, 2.303 Å; Sn2–O3, 2.243 Å) are longer than that found in catena-[(μ_2 -hydroxo)- μ_2 -dimesylamide-bis(methyl-tin)] (2.118 and 2.126 Å) [18], but large shorter than the sum of the van der Waals radii of Sn and O atoms, 3.68 Å [19]. The Sn1–S1 bond length (2.423 Å) around Sn1 atom is slight longer than that found in [(*n*-

Pr)₃NH][Me₂Sn(μ^2 -SCH₂CO₂)Cl] (2.4026 Å) [11a] and approaches the sum of the covalent radii of Sn and S atoms, 2.42 Å [19]. The distances of Sn1–O1 (2.268 Å) and Sn2–O2A (2.303 Å), in which these two Sn–O bonds are linked by the carboxylate group, show the carboxylate bond to tin in a highly symmetric fashion (the difference between them is only 0.035 Å) and are significantly shorter than the common polymeric tri- or diorganotin carboxylates [20]. The two carboxyl C–O bonds also do not differ significantly (C1–O1, 1.255 Å; C1–O2, 1.239 Å), indicating substantial delocalization of the –CO₂ π -electron density and bond to the two tin with the bidentate mode [20]. This is in well agreement with what has been found in the IR spectrum.

As shown in Fig. 2a, the polymeric structure of **1** is a 1D helical chain, which can be considered as the tri- and dimethyltin moieties are linked together by the alternate μ_2 -OH groups and [μ_3 -SCH₂CO₂]²⁻ units running along [101] direction. The ‘pitch’ (17.066 Å) of this helix is large shorter than that found in 4-[2-(triethylstannyl)-tetrazol-5-yl]pyridine (40 Å) [21], which may be due to the different molecular conformation and bonding mode between the two complexes. Interestingly, these helical chains are further linked through inter-helix O–H···S (O3–S1, 3.490 Å) H-bonding interactions to form a 2D corrugated sheet (Fig. 2b), where the loose cavities provided by these H-bonding interactions are of 16.929 (S1–O3B) × 6.675 Å

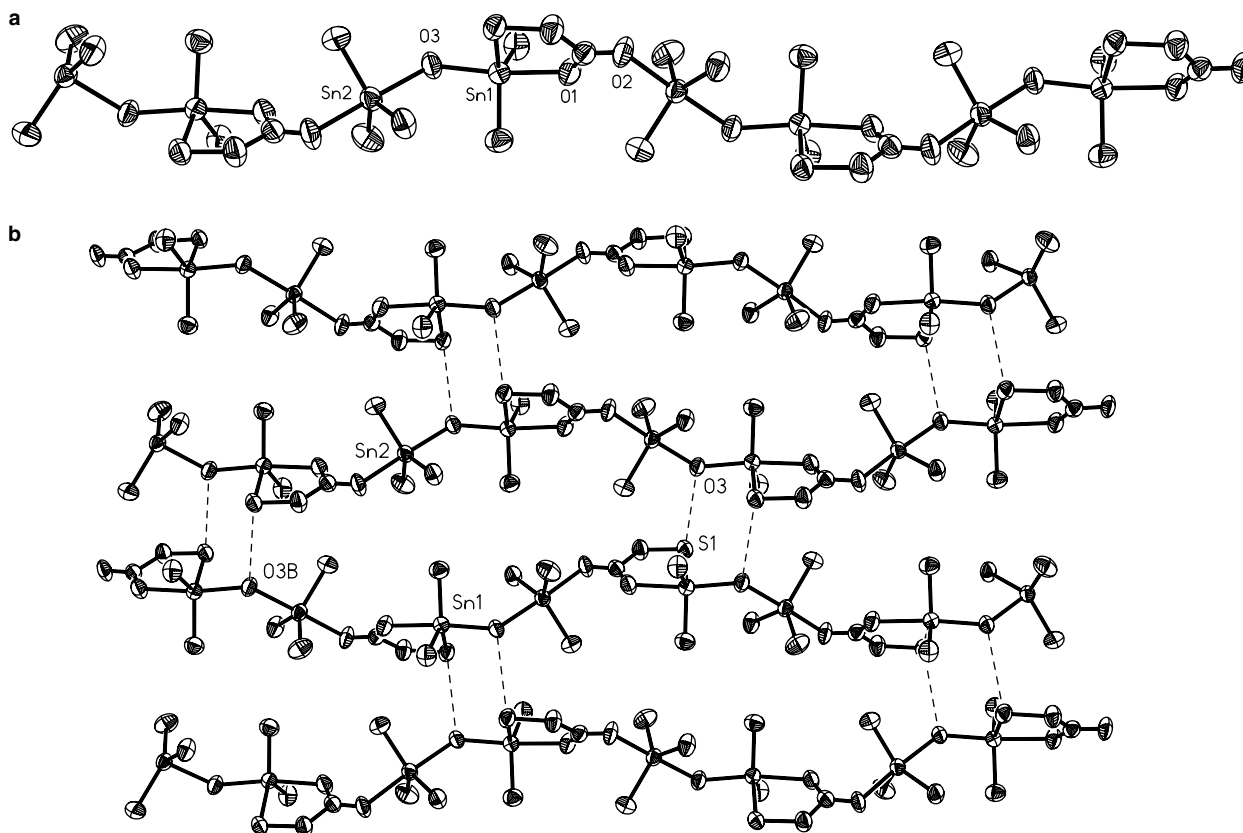


Fig. 2. (a) The 1D helical chain structure of complex **1**, which are linked by bridged hydroxyl groups and carboxyl groups. (b) These helical chains in complex **1** are linked together through inter-helix O–H···S (O3–S1, 3.490 Å) H-bonding interactions to form a 2D corrugated sheet.

(Sn1–Sn2) and are almost completely filled by the methyl groups on tin atoms that protrude into each interior. Therefore, like most of the organometal-networks, there are no solvent molecules that have been found in these cavities.

In conclusion, through the solvothermal reaction, we have obtained a novel mixed tri- and dimethyltin coordination polymer $\{[(\text{Me}_3\text{Sn})(\mu_2\text{-OH})][\text{Me}_2\text{Sn}(\mu_3\text{-SCH}_2\text{CO}_2)]\}_n$ (**1**) derived from the trimethyltin chloride and mercaptoacetic acid. This may open a new approach to prepare novel organotin complexes that hardly arise from the common synthetic methods with use of the simple organotin compounds and organic ligands as the starting materials.

3. Experimental details

3.1. Materials and measurements

Trimethyltin chloride and mercaptoacetic acid were purchased from Aldrich. Analytical grade solvents were dried before use. The melting points were obtained with Kofler micromelting point apparatus and are uncorrected. Infra-red-spectra were recorded on a Nicolet-460 spectrophotometer using KBr discs and sodium chloride optics. ^1H , ^{13}C and ^{119}Sn NMR spectra were recorded on a Varian Mercury Plus 400 spectrometer operating at 400, 101 and 186.50 MHz, respectively. The chemical shifts were given in ppm in CDCl_3 solvent. Elemental analyses were performed with a PE-2400II apparatus.

3.2. Synthesis of complex 1

A mixture of mercaptoacetic acid (0.092 g, 1 mmol), KOH (0.112 g, 2 mmol), trimethyltin chloride (0.400 g, 2 mmol), CH_3OH (10 ml) and H_2O (5 ml) was heated in a Teflon-lined autoclave at 150 °C for 3 days. After cooling down to room temperature, colorless crystals were collected and washed with methanol. Yield: 0.357 g, 85%. m.p. > 250 °C (dec.). Anal. Calc. for $\text{C}_7\text{H}_{18}\text{O}_3\text{SSn}_2$: C, 20.03; H, 4.32; Found: C, 20.05; H, 4.40%. IR (KBr, cm^{-1}): $\nu(\text{OH})$ 3050, $\nu(\text{COO})_{\text{as}}$ 1587, $\nu(\text{COO})_{\text{s}}$ 1385, $\nu(\text{Sn}-\text{C})$ 568, $\nu(\text{Sn}-\text{O})$ 461, $\nu(\text{Sn}-\text{S})$, 325. ^1H NMR (CDCl_3 , ppm): δ 0.85 (s, 9H, Sn- CH_3^{b} , $^2J_{\text{Sn}-\text{H}} = 72.8$ Hz), 0.87 (s, 6H, Sn- CH_3^{a} , $^2J_{\text{Sn}-\text{H}} = 70.5$ Hz), 3.48 (s, 2H, SCH_2), 3.85 (s, 1H, OH). ^{13}C NMR (CDCl_3 , ppm): δ -1.5 (Sn- CH_3^{b} , $^1J_{\text{Sn}-\text{C}} = 492.0$ Hz), -0.9 (CH_3 , Sn- CH_3^{a} , $^1J_{\text{Sn}-\text{C}} = 477.5$ Hz), 37.0 (CH_2S), 176.8 (COO) (a and b represent signals due to Sn- Me_2 and Sn- Me_3 , respectively). ^{119}Sn NMR (CDCl_3 , ppm): δ -128.6, -195.5.

3.3. X-ray structures determination of complex 1

Crystal data for $\text{C}_7\text{H}_{18}\text{O}_3\text{SSn}_2$: crystal dimensions $0.32 \times 0.21 \times 0.13$ mm³, $M = 419.65$, monoclinic, space group $P2(1)/n$, $a = 11.045(2)$ Å, $b = 12.219(2)$ Å, $c = 11.268(2)$ Å, $\beta = 114.831(2)^\circ$, $V = 1380.0(5)$ Å³, $Z = 4$, $\rho_{\text{cal}} = 2.020$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu =$

3.751 mm⁻¹, $T = 298(2)$ K, 7038 reflections collected for $2.17\text{--}25.03^\circ$, 2427 independent reflections. $R_1 = 0.0290$, $wR_2 = 0.0677$ for reflections with $I > 2\sigma(I)$.

Data collection was by means of a Bruker SMART CCD 1000 diffractometer. The structure was solved by direct-methods using SHELXS-97 and refined against F^2 by full-matrix least squares using SHELXL-97.

4. Supplementary material

Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Center as supplementary publication no. CCDC-254169. Copies of the data can be obtained free of charge on application to the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>).

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