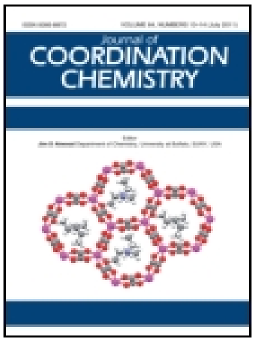

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Syntheses and characterizations of di- and tri-organotin carboxylates of 4-oxo-4-phenyl butanoic and 4-(4-biphenyl)-4-oxobutyric acid

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A family of organotin carboxylates, $[(C_6H_5)_3Sn(L^1)_2]$ (**1**) and $[(C_6H_{11})_2Sn(L^2)]$ (**2**), was synthesized from reaction of R_3SnOH and R'_2SnO (where $R = \text{phenyl}$, $R' = \text{cyclohexyl}$) with 4-oxo-4-phenyl butanoic (L^1H) and 4-(4-biphenyl)-4-oxobutyric acid (L^2H) in suitable mole ratios. They have been characterized by elemental analysis, FT-IR, ^1H NMR spectroscopy, and single-crystal X-ray diffraction analysis. In **1**, the geometry of tin is trigonal bipyramidal. Supramolecular architecture of **1** shows a polymeric chain structure with Ph_3Sn centers linked by $PhCOC_2H_4CO_2$ ligands *via* $O \rightarrow Sn$ interactions. $Sn \cdots Sn$ non-bonded distances in the repeating 1-D polymeric structure equal 5.835 Å. Complex **2** contains an angular sector and adopts a distorted tetrahedral geometry.

Keywords: Organotin carboxylate; 4-Oxo-4-phenyl butanoic; 4-(4-Biphenyl)-4-oxobutyric acid; Synthesis; Crystal structure

1. Introduction

Chemical structures and biological activities of organotin carboxylate compounds have attracted attention for their antitumor activity, antineoplastic, antituberculosis agents, diversity of structures, and industrial and agricultural applications [1–5].

Various organotin carboxylates can be obtained, such as monomers, dimers, tetramers, oligomeric ladders, and hexameric drums, utilizing different stoichiometries of organotin and the carboxylic acid [6]. Antifungal and antitumor activities of organotin complexes are influenced by the coordination number of tin and the structure

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of the molecule [7]. The 4-(4-biphenyl)-4-oxobutyric acid (Fenbufen) is a non-steroidal anti-inflammatory drug used in the treatment of inflammations such as osteoarthritis, ankylosing spondylitis, and tendinitis. It is also an antipyretic. Therapeutic properties of Fenbufen can be attributed to its inhibiting action to cyclooxygenase which is responsible for the biosynthesis of prostaglandins. Tian and co-workers carried out elegant work on some organotin carboxylates by the use of Fenbufen as ligand [8]. However, dicyclohexyltin ester based on Fenbufen (L^2H) has not been reported. We recently reported syntheses of organotin carboxylates using 4-oxo-4-phenyl butanoic acid as ligand reacting with tricyclohexyltin hydroxide. Intermolecular coordination between oxygen from carbonyl groups and tin was discovered with a distance of 0.7280 nm between tins [9]. As an extension of these studies, now we report syntheses and characterizations of new organotin(IV) 4-oxo-4-phenyl butanoates, $[(C_6H_5)_3Sn(L^1)_2]$ (**1**) and $[(C_6H_{11})_2Sn(L^2)]$ (**2**) (where L^1H indicates 4-oxo-4-phenyl butanoic acid and L^2H Fenbufen). The geometry around tin in **1** is distorted trigonal bipyramidal and the polymeric chain of **1** has a non-bonded distance of [Sn1···Sn1A and Sn1···Sn1B = (5.835 Å)]. Complex **2** shows two monodentate carboxylates and the structure presents two isosceles triangles around a planar O_2SnC_2.

2. Experimental

2.1. *Materials and methods*

Biphenyl, succinic anhydride, dicyclohexyltin oxide, and triphenyltin hydroxide were purchased from commercial sources, while 4-carbonyl-4-phenyl butanoic acid (L^1H) and Fenbufen (L^2H) were prepared according to literature procedures [10]. All chemicals were used without purification. The solvents were purified before use by standard procedures. Melting points were obtained by open capillaries and are uncorrected. ^1H NMR spectra were recorded in $CDCl_3$ on a Varian Mercury 300 MHz spectrometer. Infrared (IR) spectra using KBr pellets were recorded on an Alpha Centauri FT/IR spectrometer (400–4000 cm^{-1}). Elemental analyses were carried out on a Perkin-Elmer PE 2400 CHN instrument and by gravimetric analysis for Sn [11].

Single-crystal X-ray diffraction data for **1** and **2** were recorded on a Bruker CCD diffractometer using the ω/φ scan technique with Mo-Kα radiation ($\lambda = 0.71073$ Å). Absorption corrections were applied by using multiscan techniques [12]. The structure was solved by direct methods with SHELXS-97 [13], and refined by full-matrix least squares with SHELXL-97 [14] within WINGX [15].

All non-hydrogen atoms were refined with anisotropic temperature parameters; hydrogen atoms were refined as rigid groups. Crystal data and experimental details of the structure determinations are listed in table 1.

2.2. *Syntheses*

2.2.1. Synthesis of 4-carbonyl-4-phenyl butanoic acid (L^1H). In a three-necked flask, succinic anhydride (4.003 g, 0.04 mol), anhydrous aluminum chloride (10.67 g,

Table 1. Crystal data and refinement details for **1** and **2**.

Empirical formula	$C_{28}H_{24}O_3Sn$	$C_{44}H_{48}O_6Sn$
Formula weight	527.16	791.51
Temperature (K)	296(2)	293(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	$P21/n$	$C2/c$
Unit cell dimensions (Å, °)		
a	12.8831(14)	28.417(3)
b	11.3890(12)	6.6070(7)
c	17.0873(18)	23.608(3)
α	90	90
β	104.079(2)	121.149(3)
γ	90	90
Volume ($Å^3$), Z	2431.8(4), 4	3793.4(7), 4
Calculated density ($Mg\,m^{-3}$)	1.440	1.386
Absorption coefficient (mm^{-1})	1.076	0.721
$F(000)$	1064	1064
Crystal size (mm^3)	$0.35 \times 0.31 \times 0.28$	$0.31 \times 0.27 \times 0.21$
θ range (°)	1.79–28.41	1.67–25.02
Limiting indices	$-16 \leq h \leq 10$; $-14 \leq k \leq 13$; $-22 \leq l \leq 22$	$-33 \leq h \leq 31$; $-7 \leq k \leq 7$; $-19 \leq l \leq 28$
Reflections collected/unique	14,463/5653 [$R(\text{int}) = 0.0549$]	10,285/3327 [$R(\text{int}) = 0.0204$]
Completeness to $\theta = 25.00$ (%)	99.7	99.3
Absorption correction	Semi-empirical from equivalents	Semi-empirical from equivalents
Max. and min. transmission	0.740 and 0.693	0.859 and 0.800
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	5653/6/289	3327/0/231
Goodness-of-fit on F^2	0.970	1.067
Final R indices [$I > 2\theta(I)$]	$R_1 = 0.0414$, $wR_2 = 0.0638$	$R_1 = 0.0235$, $wR_2 = 0.0569$
R indices (all data)	$R_1 = 0.0895$, $wR_2 = 0.0755$	$R_1 = 0.0261$, $wR_2 = 0.0584$
Largest difference peak and hole ($e\,Å^{-3}$)	0.405 and −0.563	0.472 and −0.178

0.08 mol), and 60 mL benzene were added. The mixture was stirred for 6 h at 50°C and transferred into a beaker. After cooling to room temperature it was hydrolyzed with aqueous HCl (20%) to obtain a white solid which was collected by filtration. The solid was then dissolved in 20% aqueous NaOH and the excess of solvent was removed by hydrodistillation. The distillate obtained was acidified by 20% HCl. The solid was precipitated, washed with water, and recrystallized from ethanol before a pure white powder was obtained. The yield was 63%; m.p. 122–124°C; IR (KBr, cm^{-1}): $\nu(O\cdots H)$ 3400–2500; $\nu_{asym}(COO)$ 1684; $\nu_{sym}(COO)$ 1399. ^1H NMR ($CDCl_3$) $\delta = 2.3$ (s, 4H, –CH_2–CO), 7.01–8.23 (m, 5H, Ar–H), 11.63 (s, 1H, –COOH).

2.2.2. Synthesis of (4-(4-biphenyl)-4-oxobutyric acid) (L^2H). L^2H was prepared by the same procedure as L^1H in which mixture of succinic anhydride (4.003 g, 0.04 mol), anhydrous aluminum chloride (10.67 g, 0.08 mol), biphenyl (6.17 g, 0.04 mol), and carbon disulfide (CS_2) 60 mL used as solvent gave a pure white powder whose yield was 63%; m.p. 122–124°C; IR (KBr, cm^{-1}): $\nu(O\cdots H)$ 3400–2500; $\nu_{asym}(COO)$ 1680; $\nu_{sym}(COO)$ 1441. ^1H NMR ($CDCl_3$) $\delta = 2.19$ (s, 4H, –CH_2–CO), 7.20–7.72 (m, 9H, Ar–H), 11.60 (s, 1H, –COOH).

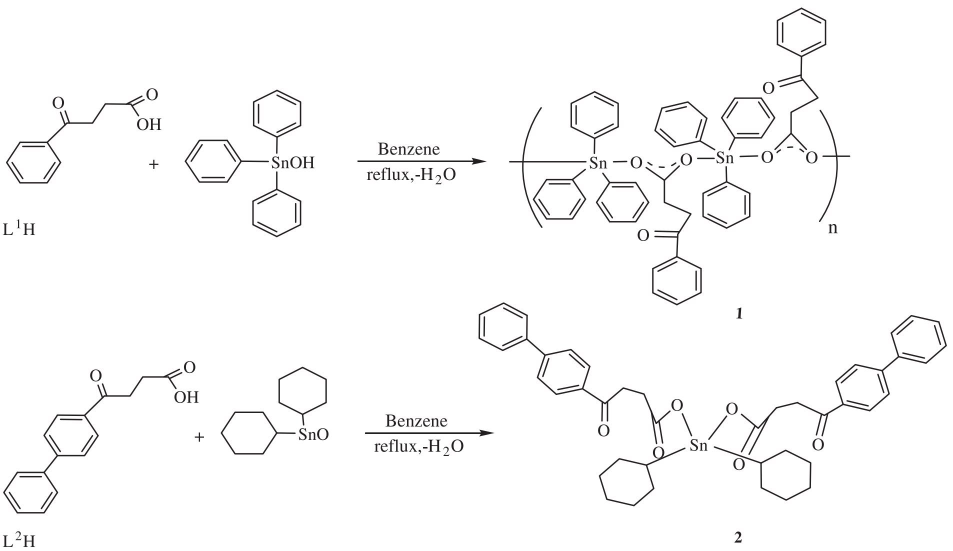
2.2.3. Synthesis of [$(C_6H_5)_3Sn(L^1)_2$] (1). Triphenyltin hydroxide (0.37 g, 1 mmol) was added to 50 mL of benzene in which L^1H (0.53 g, 1 mmol) was dissolved. After refluxing for 6 h, the binary azeotrope water/benzene was distilled off with a Dean-Stark funnel. The reaction mixture was cooled to room temperature and evaporated under vacuum. Then, recrystallizing the precipitated part twice with ethanol, **1** was obtained with yield of 70%; m.p. 130–132°C. Anal. Calcd for $C_{28}H_{24}O_3Sn$ (%): C, 63.63; H, 4.55; Sn, 22.52. Found (%): C, 63.79; H, 4.59; Sn, 22.52. IR (KBr, cm^{-1}): ν(C=O), 1684; ν_{sym}(COO) 1403, ν_{asym}(COO) 1525; ν(Sn–O) 450 cm^{-1}; ν(Sn–C) 693. ^1H NMR ($CDCl_3$, ppm) $\delta = 2.85$ (t, 2H, CH_2–COOSn); 2.87 (t, 2H, CH_2–COC_6H_5); 7.20–7.97 (m, 20H, Ar–H).

2.2.4. Synthesis of [$(C_6H_{11})_2Sn(L^2)$] (2). Similar to the procedure for **1**, dicyclohexyltin oxide (0.301 g, 1 mmol) was added to L^2H (0.25 g, 1 mmol) and **2** was obtained with yield of 66%; m.p. 180–182°C. Anal. Calcd for $C_{44}H_{48}O_6Sn$ (%): C, 66.65; H, 6.06; Sn, 14.98. Found (%): C, 66.76; H, 6.11; Sn, 15.00. IR (KBr, cm^{-1}): ν(C=O), 1684; ν_{sym}(COO) 1444, ν_{asym}(COO) 1601; ν(Sn–O) 492 cm^{-1}; ν(Sn–C) 525. ^1H NMR ($CDCl_3$, ppm) $\delta = 1.94$–1.33 (m, 22H, cyclohexyl–H, SnC_6H_{11}); $\delta = 2.87$–2.79 (t, 2H, CH_2–COOSn); 2.89–2.76 (t, 2H, CH_2–$COC_{12}H_9$); 7.20–7.70 (m, 18H, Ar–H).

3. Results and discussion

3.1. *Syntheses*

Proposed schematic diagrams of syntheses of **1** and **2** are shown in scheme 1.



Scheme 1. Syntheses of **1** and **2**.

3.2. *IR spectroscopic studies*

In the IR spectra of L^1H and L^2H, the O–H stretching vibration was at 3450 and 2800 cm^{-1}. After interactions between carboxyl group and tin, the O–H absorption disappeared, and ν_{as}(COO) and ν_{sym}(COO) showed bathochromic shift [16]. The difference of $\Delta\nu(\nu_{as}(COO)–\nu_{sym}(COO))$ is used to determine the coordination mode of carboxylate tin complexes; if $\Delta\nu$ is below 200 cm^{-1}, the carboxyl group is bidentate and if $\Delta\nu$ is more than 200 cm^{-1}, the carboxyl is unidentate [17, 18]. The difference of $\Delta\nu$ for **1** and **2** are 285 and 239 cm^{-1}, respectively. The carboxyl groups in **1** and **2** are unidentate [19, 20].

Absorptions for **1** at 1684, 693, and 450 cm^{-1} can be assigned to ν(acyl C=O), ν(Sn–C), and (Sn–O), respectively. In **2**, 525 and 492 cm^{-1} are assigned to ν(Sn–C) and (Sn–O) stretching modes.

3.3. *^{1}H NMR spectra*

In the ^{1}H NMR spectra of **1** and **2**, δCOO–H disappeared, proving that the carboxyl group participates in coordination at tin. In **1**, absorption of two types of protons in ethyl are 2.85 and 2.87 ppm, while in **2**, the chemical shifts of methylene proton in cyclohexyl group are 1.94–1.33 and 7.20–7.70 ppm for protons in aryl groups. The hydrogen protons of CO–CH_2–CH_2–COOSn in **2** are at 2.87–2.79 and 2.89–2.76 ppm.

3.4. *Crystal structures of 1 and 2*

The crystal structures, 1-D polymeric chain, and the fragment unit of **1** and **2** are shown in figures 1–4; selected bond lengths and angles are given in table 2.

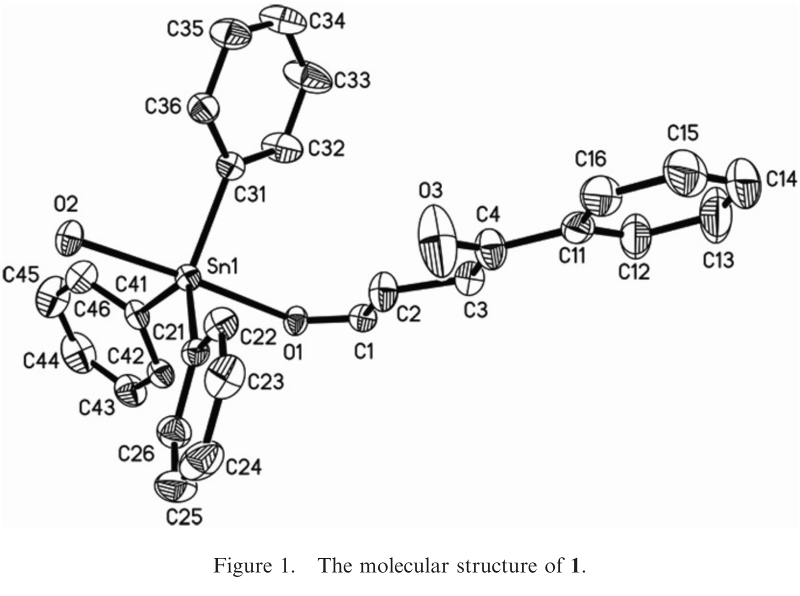


Figure 1. The molecular structure of **1**.

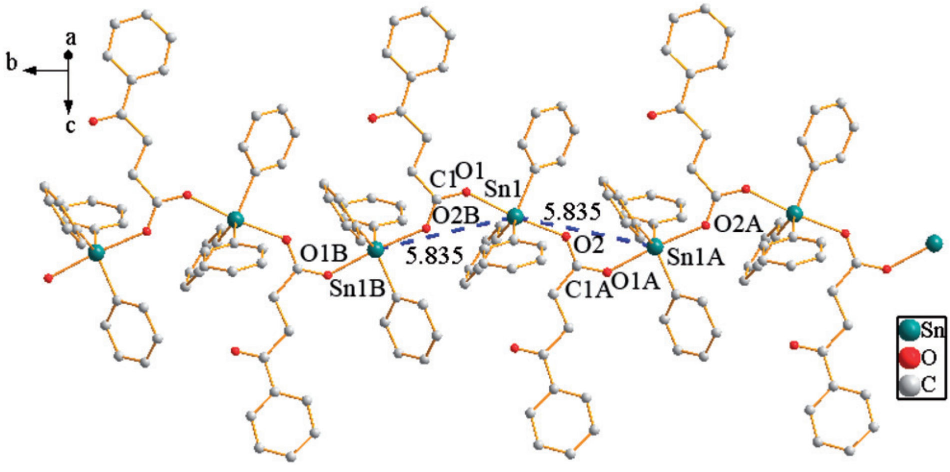


Figure 2. Perspective view of polymeric chain formed by intermolecular O → Sn interactions. Hydrogen atoms are omitted for clarity. Thermal ellipsoids are drawn at 30% probability level. Sn1···Sn1A or Sn1···Sn1B = 5.835 Å (the non-bonded distances). Symmetry codes: (A) $5/2-x$, $y-1/2$, $1/2-z$; (B) x, $-1-y$, z.

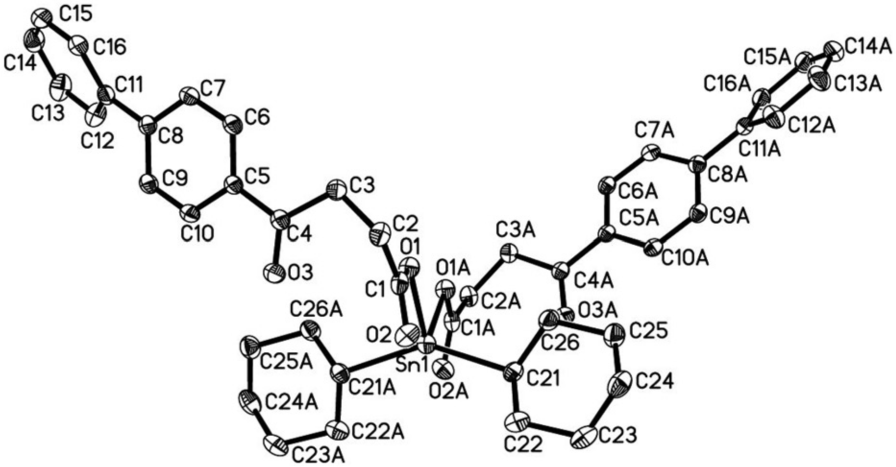


Figure 3. The molecular structure of **2**. Symmetry codes: (A) $-1-x$, y, $1/2-z$.

3.4.1. Structure of 1. In **1**, tin is five-coordinate and a distorted *trans*-O_2SnC_3 trigonal-bipyramidal structure by coordinating an addition oxygen O(2), which can be regarded as a ligand from carboxylate anions C1–O2#2. The equatorial plane is defined by the three R groups, while axial positions are occupied by O1 and O2. The Sn–C bonds are 2.123(3)–2.128(3) Å. The carboxylate anions O1–C1 and C1–O2#2 linked to Sn1 give non-equal Sn–O bond distances of [Sn1–O1 = 2.261(2) Å and Sn1–O2 = 2.244(2) Å], respectively. Thus, O2B forms a closer contact with Sn1 than O1, which opens up an obtuse angle [O2#2–C1–O1 to 123.5(3)°] from the ideal of 90°. The axial angle O2–Sn1–O1 = 174.23(8)° is close to linear. In the polymeric chain formed, O2 of carboxylate functions as a bridge, connecting tin centers in a series of intermolecular O → Sn

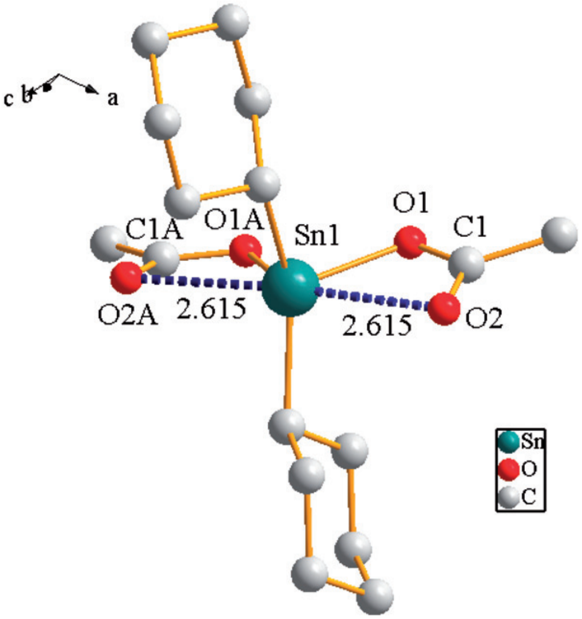


Figure 4. View of the tetrahedral tin in **2**, showing Sn1···O interactions. Symmetry codes: (A) $-1-x$, y, $1/2-z$.

Table 2. Selected bond lengths (Å) and angles (°) of **1** and **2**.

1			
Sn1–C21	2.123(3)	O1–C1	1.266(4)
Sn1–C31	2.126(3)	O2–C1$^{\#1}$	1.254(4)
Sn1–C41	2.128(3)	O3–C4	1.204(5)
Sn1–O2	2.244(2)	C1–O2$^{\#2}$	1.254(4)
Sn1–O1	2.261(2)	C1–C2	1.508(5)
C21–Sn1–C31	122.14(13)	C41–Sn1–O1	88.15(11)
C21–Sn1–C41	120.35(14)	O2–Sn1–O1	174.23(8)
C31–Sn1–C41	117.49(13)	C1–O1–Sn1	131.9(2)
C21–Sn1–O2	90.57(11)	C1$^{\#1}$–O2–Sn1	137.9(2)
C31–Sn1–O2	92.02(12)	O2$^{\#2}$–C1–O1	123.5(3)
C21–Sn1–O1	91.81(11)	O1–C1–C2	118.9(3)
C31–Sn1–O1	91.16(12)	C1–C2–C3	115.7(3)
2			
Sn1–O1$^{\#1}$	2.1063(13)	O2–C1	1.233(2)
Sn1–O1	2.1063(13)	O3–C4	1.217(2)
Sn1–C21$^{\#1}$	2.1412(19)	C1–O1–Sn1	103.29(12)
Sn1–C21	2.1412(19)	O2–C1–O1	120.82(18)
O1–C1	1.300(2)	O2–C1–C2	122.69(18)
O1$^{\#1}$–Sn1–O1	81.98(7)	O1–C1–C2	116.48(18)
O1$^{\#1}$–Sn1–C21$^{\#1}$	108.43(6)		
O1–Sn1–C21$^{\#1}$	99.89(6)		
O1$^{\#1}$–Sn1–C21	99.89(6)		
O1–Sn1–C21	108.43(6)		
C2$^{\#1}$–Sn1–C21	142.29(11)		

Symmetry transformations used to generate equivalent atoms: for **1**: $^{\#1}$ $-x+5/2$, $y+1/2$, $-z-1/2$; $^{\#2}$ $-x+5/2$, $y-1/2$, $-z-1/2$, for **2**: $^{\#1}-x-1$, y, $-z+1/2$.

interactions (scheme 1), leading to 1-D polymeric chains (figure 2) containing equal non-bonded distances of [Sn1···Sn1A and Sn1···Sn1B = (5.835 Å)]. Two adjacent tins are separated by three atoms. Distance reported of two adjacent tins is from 0.519 ± 0.021 nm to 0.7280 nm, which indicates non-covalent character in the solid state. Substituents on Sn are not influential in coordination with carboxylate [21, 22]. A structure ($Bu_3SnO_2CCH = CHC_6F_3$) has been reported [23] in which similar polymer is found, with a repeat chain connected by O → Sn and separated by O–C–O distance of 5.185 Å.

3.4.2. Structure of 2. The molecular structure of **2** is centrosymmetric; tin is four-coordinate with a distorted tetrahedral geometry (figure 3). This coordination is completed by two monodentate carboxylates *via* O1 and O1A. Tetrahedral geometry is defined by Sn1, C21, and C21A of cyclohexyls and O1 and O1A. O1, Sn1, and O1A and C21, Sn1, and C21A are isosceles triangles forming an angular sector with Sn1 as vertex. Sn1–C21 and Sn1–C21A are 2.1412(19) Å, Sn1–O1A and Sn1–O1 = 2.1063 Å and the angles are C21–Sn1–C21A = 142.29 Å and O1A–Sn1–O1 = 81.98 Å. Important extension angle at 142.29 Å may be due to bulky butyl groups attached to Sn. The Sn1···O interactions in **2** are shown in figure 4. Rotation from planarity for this structure can be visualized. O2A, C1A, C2A, O1A, and Sn1 are coplanar; the bond lengths of Sn1···O2 and Sn1···O2A = 2.615 Å are less than the sum of the van der Waals radii (3.58 Å) [24].

4. Conclusion

We have synthesized and characterized two organotin carboxylates based on the esterification of 4-oxo-4-phenyl butanoic acid and 4-(4-biphenyl)-4-oxobutyric acid with the corresponding triorganotin hydroxide and oxide in 1 : 1 molar ratio. The two complexes have monodentate carboxylates and **1** is a polymeric chain in which two adjacent tins are separated by three atoms. Sn···Sn distance is 5.835 Å. In **2**, Sn is at the vertex of two isosceles triangles, with two monodentate carboxylates. Further studies on the biological activities of **1** and **2** are recommended.

Supplementary material

CCDC nos 791273 and 799845 contain the supplementary crystallographic data for **1** and **2**. The data can be obtained free of charge from the Cambridge Crystallographic Data Center *via* www.ccdc.cam.ac.uk/data_request.cif

Acknowledgments

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