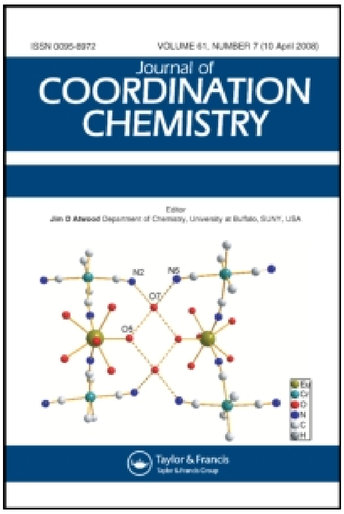

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Synthesis, crystal structure, and characterization of dimeric tetraorganodistannoxane and two tricyclohexyltin carboxylates

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Synthesis, crystal structure, and characterization of dimeric tetraorganodistannoxane and two tricyclohexyltin carboxylates

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Three diorganotin(IV) series and triorganotin(IV) complexes, $[(C_6H_{11})_2Sn]_4(L_1)_2O_2(OH)_2$ (**1**), $(C_6H_{11})_3Sn(HL_2)$ (**2**), and $(C_6H_{11})_3SnL_3$ (**3**) (where HL_1 is 2-(4-isopropyl benzoyl) benzoic acid, H_2L_2 is phthalic acid and HL_3 is 2-benzoyl benzoic acid), were synthesized and their crystal structures were determined. There are four crystallographically unique Sn centers in the structure of **1**, which consists of a $Sn_4O_2(OH)_2$ ladder unit, and the ladder consists of four tins held together by two μ_3-oxygens and two μ_2-oxygens. The supermolecular motif of **1** is a 2-D structure linked by $O–H \cdots O$ hydrogen bonds. The asymmetric unit of **2** contains two crystallographically independent monomers. The supramolecular architecture of **2** is a 2-D layer structure linked by face-to-face π–π interactions between phenyl rings of adjacent L_2 anions. The structure of **3** contains one tricyclohexyltin cation and one L_3 anion. The $Sn \cdots O$ interactions lead the whole structure to a supramolecular chain. Elemental analysis, infrared, and ^1H NMR of **1**–**3** were investigated and discussed.

Keywords: Organotin; Tetraorganodistannoxane; Synthesis; Crystal structure; Coordination polymer

1. Introduction

Organotin carboxylate derivatives have antitumor activity, rich structural chemistry [1], and wide use as PVC stabilizers, as catalysts, and in polyurethane polymerization [2]. The structural studies of organotin(IV) compounds of carboxylic acids are of growing interest because there are many possible bonding interactions between oxygen of carboxyl and tin. Studies on diorganotin(IV) or triorganotin(IV) compounds of carboxylic acid [3] revealed new structural types which may lead to compounds with different activities. However, dicyclohexyltin and tricyclohexyltin carboxylates have not been reported [4]. We recently reported some organotin compounds with different carboxylates. As the continuation of this investigation, we have now synthesized three organotin carboxylates: $[(C_6H_{11})_2Sn]_4(L_1)_2O_2(OH)_2$ (**1**), $(C_6H_{11})_3Sn(HL_2)$ (**2**), and $(C_6H_{11})_3SnL_3$ (**3**) ($HL_1 = $ 2-(4-isopropyl benzoyl) benzoic acid; $H_2L_2 = $ phthalic acid; $HL_3 = $ 2-benzoyl benzoic acid). The ^1H NMR and FT-IR spectroscopies characterize **1**–**3**.

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2. Experimental

2.1. *Materials and methods*

Phthalic acid (HL_2) was purchased from commercial sources and used without purification; the solvents were purified before use by standard procedures. The melting points were obtained in open capillaries and are uncorrected. Elemental analyses were performed with a Perkin-Elmer PE 2400 CHN instrument and gravimetric analysis was performed for Sn. ^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 FI/IR spectrometer (400–4000 cm^{-1}).

X-ray crystallographic data for **1**–**3** were recorded on a Bruker CCD Area detector image plate diffractometer using the ω/φ scan technique with Mo-Kα radiation ($\lambda = 0.71073$ Å). Absorption corrections were applied using multi-scan techniques [5]. The structures were solved by direct methods with SHELXS-97 [4] and refined by full-matrix least squares with SHELXL-97 [6] within WINGX [7]. All non-hydrogen atoms were refined with anisotropic temperature parameters. Hydrogens were refined as rigid groups. The detailed crystallographic data and structure refinement parameters for **1**–**3** are summarized in table 1.

2.2. *Syntheses*

2.2.1. Synthesis of 2-(4-isopropylbenzoyl) acid (HL_1). Phthalic anhydride (5.92 g, 0.04 mol), anhydrous aluminum chloride (10.67 g, 0.08 mol), and dry isopropyl benzene (60 mL) were added to a three-neck flask. 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 dissolved in 20% aqueous NaOH and the excess solvent was removed by hydrodistillation. The distillate obtained was acidified with 20% HCl and the solid got precipitated. Then the filtrate was washed with water; the solid thus obtained was recrystallized from the ethanol before a pure white powder was obtained. The yield was 63%, m.p. 118–120°C, IR (KBr, cm^{-1}): ν_{sym}(COO) 1420, 1569; ν_{asym}(COO) 1599, 1614; ν(O–H) 3440; ^1H NMR ($CDCl_3$) δ: 1,24 (d, 6H, –CH_3), 3.05 (m, 1H, –CH), 7.26–8.11. (m, 8H, Ar–H), 11.63 (s, 1H, –COOH). Anal. Calcd for $C_{17}H_{16}O_3$ (%): C, 76.10; H, 6.01. Found (%): C, 76.03; H, 5.96.

2.2.2. Synthesis of 2-benzoyl benzoic acid (HL_3). HL_3 was prepared by the same procedure as HL_1; in which the mixture of phthalic anhydride (5.92 g, 0.04 mol), anhydrous aluminum chloride (10.67 g, 0.08 mol), and dry benzene (60 mL) gave a pure white powder whose yield was 58%, m.p. 138–140°C, IR (KBr, cm^{-1}): ν_{sym}(COO) 1413, 1555; ν_{as}(COO) 1584, 1606; ν(O–H) 3445; ^1H NMR ($CDCl_3$) δ: 7.26–8.13 (m, 9H, Ar–H), 11.13 (s, 1H, –COOH). Anal. Calcd for $C_{14}H_{10}O_3$ (%): C, 74.33; H, 4.46. Found (%): C, 74.26; H, 4.42.

2.2.3. Synthesis of $[(C_6H_{11})_2Sn]_4(L_1)_2O_2(OH)_2]$ (1). A mixture of dicyclohexyltin oxide (0.301 g, 1 mmol) and HL_1 (0.268 g, 1 mmol) in dry benzene (40 mL) was refluxed

Table 1. Crystal data and details of structure refinement parameters for **1**–**3**.

	1	**2**	**3**
Empirical formula	$C_{82}H_{120}O_{10}Sn_4$	$C_{20}H_{37}O_4Sn$	$C_{32}H_{42}O_3Sn$
Formula weight	1740.54	532.25	593.35
Crystal size (mm^3)	$0.33 \times 0.24 \times 0.21$	$0.36 \times 0.30 \times 0.28$	$0.32 \times 0.27 \times 0.23$
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/c$	$P2_1/n$
Unit cell dimensions (Å, °)			
a	12.4789(11)	19.1159(11)	13.966(2)
b	17.2594(15)	15.0730(9)	14.399(2)
c	19.6118(17)	17.9044(10)	14.606(2)
β	100.7130(10)	106.1730(10)	93.101(2)
Volume (Å^3), Z	4150.3(6), 2	4954.7(5), 8	2932.8(8), 4
Calculated density ($g\,cm^{-3}$)	1.393	1.427	1.344
Absorption coefficient (mm^{-1})	1.242	1.060	0.900
$F(000)$	1784	2200	1232
θ range for data collection (°)	1.06–24.67	1.75–26.04	1.99–25.01
Reflections collected	20,527	27,313	14,618
Unique reflections	13,272 [$R_{(int)} = 0.0467$]	9750 [$R_{(int)} = 0.0361$]	5144 [$R_{(int)} = 0.0543$]
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0567$, $wR_2 = 0.1018$	$R_1 = 0.0369$, $wR_2 = 0.0812$	$R_1 = 0.0476$, $wR_2 = 0.0986$
R indices (all data)	$R_1 = 0.1288$, $wR_2 = 0.1276$	$R_1 = 0.0611$, $wR_2 = 0.0913$	$R_1 = 0.0975$, $wR_2 = 0.1164$
Goodness-of-fit on F^2	0.980	1.016	1.001
Largest difference peak and hole (e Å^{-3})	0.493 and −0.413	1.010 and −0.446	0.521 and −0480

for 8 h in a Dean–Stark separator. After cooling to room temperature, the solvent was gradually removed by evaporation under vacuum until solid product was obtained. The resulting solid was then recrystallized from ethanol/water to give buff crystals of **1**. The yield was 67%, m.p. 188–190°C. IR (KBr, cm^{-1}): ν(C–H) 2935, 2920, 2864; ν(Sn–O–Sn) 482, 422; ν(Sn–C) 541; ν_{sym}(COO) 1376, 1432; ν_{as}(COO) 1588, 1650. ^1H NMR ($CDCl_3$) δ: 1.23–1.94 (m, 88H, CH_2, SnC_6H_{11}, 12H, $-CH_3$), 2.04 (s, 2H, Sn–OH–Sn), 3.1 (m, 2H, –CH), 7.15–7.65 (m, 16H, Ph). Anal. Calcd for $C_{82}H_{120}O_{10}Sn_4$ (%): C, 56.58; H, 6.95; Sn, 27.28. Found (%): C, 56.53; H, 6.89; Sn, 27.24.

2.2.4. Synthesis of $(C_6H_{11})_3Sn(HL_2)$ (2). Complex **2** was prepared by the same procedure as **1** in which a mixture of tricyclohexyltin hydroxide (0.385 g, 1 mmol) and phthalic acid (H_2L_2) (0.166 g, 1 mmol) was refluxed for 8 h in a Dean–Stark separator. After cooling to room temperature, the solvent was gradually removed by evaporation under vacuum until solid product was obtained. The resulting solid was then recrystallized from ethanol/water to give buff crystals of **2** in 58% yield, m.p. 170–172°C. IR (KBr, cm^{-1}): ν(Sn–O–Sn) 455, 416; ν(Sn–C) 549; ν_{sym}(COO) 1351, 1443; ν_{as}(COO) 1590, 1663. ^1H NMR ($CDCl_3$) δ: 1.25–1.96 (m, 33H, CH_2, SnC_6H_{11}), 7.19–7.96 (m, 4H, Ar–H). Anal. Calcd for $C_{26}H_{37}O_4Sn$ (%): C, 58.66; H, 7.01; Sn, 22.30. Found (%): C, 58.57; H, 7.11; Sn, 22.35.

2.2.5. Synthesis of ($(C_6H_{11})SnL_3$) (3). Complex **3** was prepared by the same procedure as **1** using a mixture of tricyclohexyltin hydroxide (0.385 g, 1 mmol) and HL_3 (0.226 g, 1 mmol) in 53% yield, m.p. 138–140°C. IR (KBr, cm^{-1}): ν(Sn–O–Sn) 454, 412, ν(Sn–C) 634; ν_{sym}(COO) 1350, 1440; ν_{as}(COO) 1584, 1663. ^1H NMR ($CDCl_3$) δ: 1.23–1.93 (m, 33H, CH_2, SnC_6H_{11}), 7.26–7.76 (m, 9H, Ar–H). Anal. Calcd for $C_{32}H_{42}O_3Sn$ (%): C, 64.77; H, 7.13; Sn, 20.00. Found (%): C, 64.72; H, 7.07; Sn, 20.08.

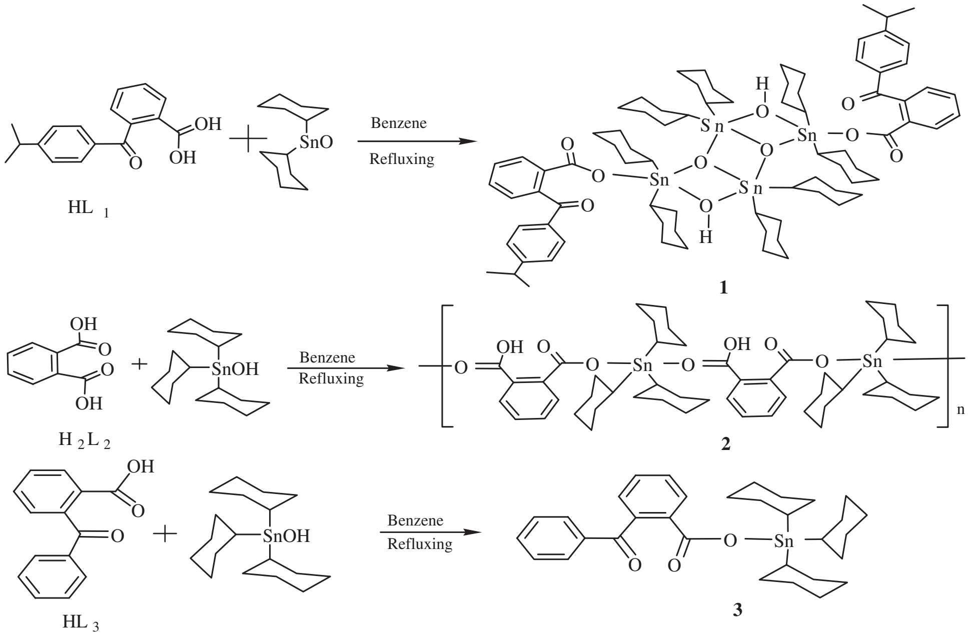
3. Results and discussion

3.1. *Syntheses*

The synthetic procedures are shown in scheme 1.

3.2. *IR spectra*

In the IR spectra of **1**–**3**, the absence of a broad band at 2500–3550 cm^{-1}, which appears in the spectra of the free ligand as ν(O–H), indicates the formation of metal–ligand bonds. The difference of $\Delta\nu(\nu_{as}(COO) - \nu_{sym}(COO))$ is used to determine the nature of bonding of tin carboxylate complexes [8]. This difference in $\Delta\nu$ between asymmetric and symmetric absorption frequencies below 200 cm^{-1} indicates that the carboxylate is bidentate, while greater than 200 cm^{-1} indicates unidentate carboxylate [9]. The ν_{as}(COO) and ν_{sym}(COO) vibrations are 1588, 1650 and 1376, 1432 cm^{-1} for **1**; 1590,



Scheme 1. The synthesis procedure of **1**–**3**.

1663 and 1351, 1443 cm^{-1} for **2**; and 1584, 1663 and 1350, 1440 cm^{-1} for **3**. The differences of $\Delta[\nu_{as}(COO) - \nu_{sym}(COO)]$ between these frequencies for **1**–**3** are close to those found for a unidentate carboxylate (218 and 212 cm^{-1} for **1**; 239 and 220 cm^{-1} for **2**; and 208 and 223 cm^{-1} for **3**), which was confirmed by X-ray structural analyses. Bands at 477 and 419 cm^{-1} are assigned to ν(Sn–O–Sn), indicating the formation of Sn–O–Sn bridges [9]. The absorption bands at 536, 585, and 549 cm^{-1} for **1**–**3** are assigned to ν(Sn–C) stretching modes [10].

3.3. *^{1}H NMR spectra*

In ^{1}H NMR spectra of the free ligands, single resonances are observed at 11–12 ppm, which are absent when the carboxylate coordinates with Sn. ^{1}H NMR of **1**–**3** show a multiplet at 1.23–1.96 ppm due to the cyclohexyl protons, and the protons on the phenyls of **1**–**3** show multiplets at 7.15–7.96 ppm.

3.4. *Crystal structures*

Detailed crystallographic data and structural refinement parameters for **1**–**3** are summarized in table 1 with selected bond lengths and angles in table 2.

3.4.1. Structure of [$(Cy_2Sn)_4(L_1)_2)O_2(OH)_2$]. As shown in figure 1, the structure of **1** consists of a $Sn_4O_2(OH)_2$ ladder unit. Four tin atoms are almost coplanar, with the largest deviation from the $Sn_4O_2(OH)_2$ ladder plane being 0.0149 Å. Each ladder consists of four tin centers held together by two μ_3-oxygens. According to their coordination environments, the four tins can be divided into two types. Sn1 and Sn4, bonded to one μ_3-oxygen, one μ_2-oxygen, and one oxygen derived from the carboxylate

Table 2. Selected bond lengths (Å) and angles (°) for **1**–**3**.

1			
Sn1–O5	2.171(9)	Sn3–O3	2.070(7)
Sn2–O2	2.024(8)	Sn3–O2	2.180(8)
Sn2–O3	2.130(8)	Sn4–O7	2.197(9)
O5–Sn1–O1	155.4(3)	O4–Sn3–O2	146.4(3)
O2–Sn1–C41	113.4(6)	O3–Sn3–C81	118.2(5)
O2–Sn1–C51	112.6(6)	O3–Sn3–C91	121.4(6)
C41–Sn1–C51	131.8(8)	C81–Sn3–C91	120.5(7)
O1–Sn2–O3	147.7(3)	O7–Sn4–O4	155.1(3)
O2–Sn2–C61	122.9(5)	O3–Sn4–C101	117.7(5)
O2–Sn2–C71	115.3(6)	O3–Sn4–C111	110.0(6)
C61–Sn2–C71	121.6(7)	C101–Sn4–C111	130.4(7)
2			
Sn1–C41	2.149(3)	Sn1–O1	2.479(2)
Sn2–C71	2.162(3)	Sn2–O7$^{\#3}$	2.284(2)
O3–Sn1–O1	171.74(8)	O7$^{\#3}$–Sn2–O5	171.60(9)
3			
Sn1–O1	2.132(4)	Sn1–C31	2.150(7)
Sn1–C41	2.144(5)		
O1–Sn1–C31	93.0(3)	O2–C1–O1	123.2(6)

Symmetry code: #3 $x, -y+5/2, z+1/2$.

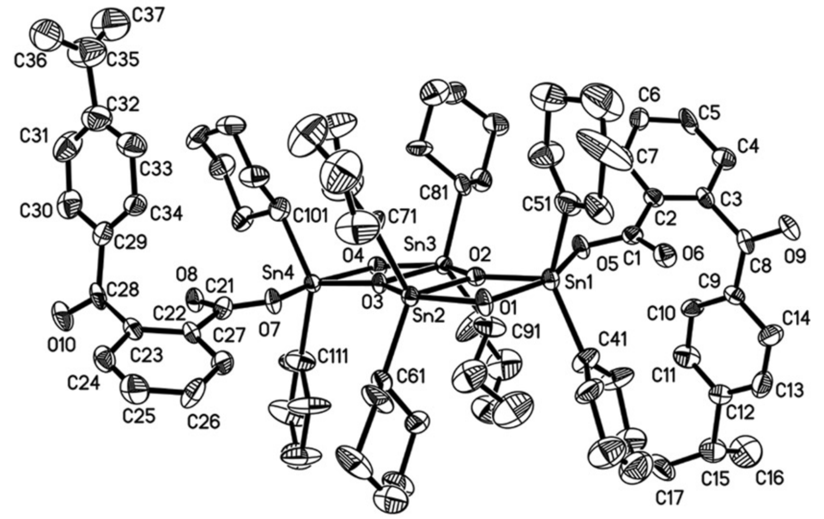


Figure 1. A view of the molecular structure of **1** showing the atomic numbering scheme. Hydrogen atoms have been omitted for clarity.

HL$_1$, can be regarded as type 1; Sn2 and Sn3, bonded to two μ_3-oxygens and one μ_3-oxygen, can be regarded as type 2. All of the tin atoms are five-coordinate with two cyclohexyl groups and three oxygens, resulting in a *cis*-$(C_6H_{11})_2SnO_3$ trigonal–bipyramidal coordination environment with two cyclohexyl groups and one oxygen in equatorial positions. The angle range of O–Sn–O is 146.4(3)–155.4(3)°, deviating considerably from 180°, which indicates that the structure is distorted. For Sn1, the trigonal plane is defined by C41, C51, and O2 with axial positions occupied by O5 and O1; the trigonal plane for Sn2 is defined by C61, C71, and O2 with axial positions occupied by O3 and O1. For Sn3, C81, C91, and O3 are equatorial and O4 and O2 are axial. For Sn4, C111, C101, and O3 are equatorial and O7 and O4 are axial. Each exocyclic Sn is also coordinated by monodentate carboxylate Sn1–O5 = 2.171(9) Å and Sn4–O7 = 2.197(9) Å.

Complex **1** displays a ladder-type structure that is analogous to the structure of symmetric distannoxanes [11]. The central cyclic four-membered Sn_2O_2 core of **1** is linked to two terminal Cy_2Sn entities through μ_3-oxos O2 and O3, and Sn–O bond lengths are Sn2–O2 = 2.024(8) Å; Sn3–O2 = 2.180(8) Å; Sn2–O3 = 2.130(8) Å; and Sn3–O3 = 2.070(7) Å. The four tin centers are held together by μ_3 O2 and O3. Sn2 and Sn3 are each bonded to two μ_3-oxos and one μ_2-oxo. Sn1 and Sn4 are each bonded to one μ_3-oxo and one μ_2-oxo, and are asymmetrically bridged by HL$_1$ through O5 and O7. The central Sn_2O_2 core is linked to the two outer Sn_2O_2 rings resulting in a ladder-type structure as shown in figures 1 and 2. The two oxygens of the central cyclic unit are tridentate, linking three Sn atoms (two endocyclic and one exocyclic). O6 and O8 of the carboxylate have weak interactions with Sn; the bond lengths of Sn1 $\cdots$ O6 (2.988 Å) and Sn4 $\cdots$ O8 (3.090 Å) are less than the sum of the Van der Waals radii (3.58 Å) [12] (figure 3). Weak O–H $\cdots$ O interactions exist between hydroxy hydrogen and carboxylate oxygen (O(1)–H(1) $\cdots$ O(10)#1 of 2.06 Å and O(4)–H(4) $\cdots$ O(9)#2 of 2.35 Å). These interactions lead **1** to form a 2-D supramolecular framework (figure 4). Detailed O–H $\cdots$ O hydrogen bond parameters are listed in table 3.

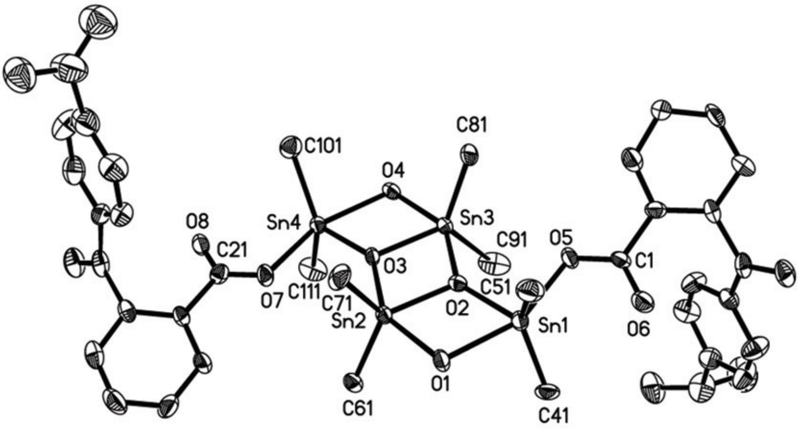


Figure 2. Repeating fragment ladder-type structure in the polymeric layer of **1**. For clarity, only the first carbons of the cyclohexyl linked to Sn are shown.

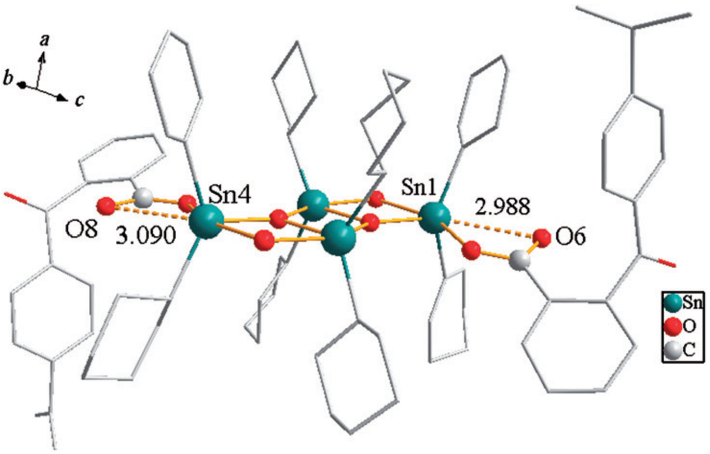


Figure 3. Repeating unit of **1**, including Sn ··· O interactions shown by orange dotted lines. Selected bond distances: [Sn1 ··· O6 = 2.988 Å and Sn4 ··· O8 = 3.090 Å].

3.4.2. Molecular and crystal structure of 2. The structure of **2** (figure 5) contains two independent units with similar conformations and only small differences in bond lengths and angles (table 2). Each unit contains two different carboxylates, one L_2 anion and one benzoic anion. The tins form distorted *trans*-O_2SnC_3 trigonal bipyramidal with three cyclohexyl groups in the equatorial plane [13]. Sn–C distances lie in the range 2.149(3)–2.162(3) Å and Sn–O bond lengths involving carboxyl oxygen are 2.284(2)–2.479(2) Å [Sn1–O1 = 2.479(2) Å; Sn1–O3 = 2.242(2) Å; Sn2–O5 = 2.339(2) Å; and Sn2–O7#3 = 2.284(2) Å]. Weak distortion angle observed can be attributed to the presence of steric interactions by three cyclohexyl groups attached to Sn1 and Sn2 (shown by the first carbon of the cyclohexyl groups), giving angles of O3–Sn1–O1 and O7#3–Sn2–O5 [171.74(8)° and 171.60(9)°, respectively]. The carboxylate oxygens of L_2 anions and benzoic anions are monodentate. Tricyclohexyltin groups are linked

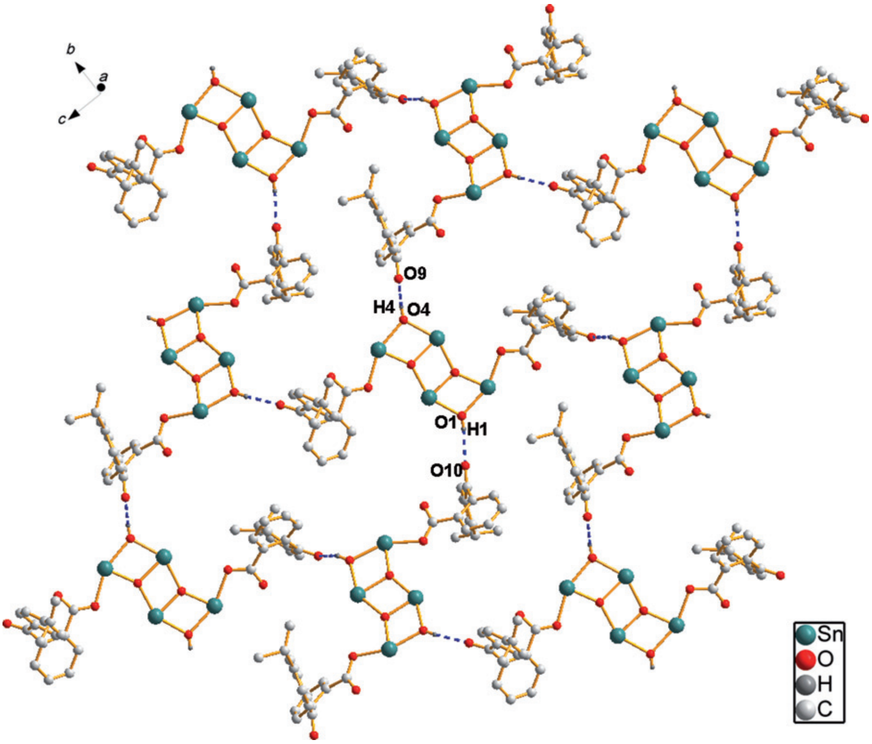


Figure 4. The 2-D network connected by intermolecular O–H···O interactions. The hydrogen atoms (except H1 and H4) and cyclohexyl groups have been omitted for clarity.

Table 3. Hydrogen bonds for **1**.

D–H···A	D–H (Å)	H···A (Å)	D···A (Å)	D–H···A (°)
O(1)–H(1)···O(10)#1	0.75	2.06	2.922(12)	169.8
O(4)–H(4)···O(9)#2	0.76	2.35	2.809(13)	133.7

#1: $-x+1$, $y-1/2$, $-z-1$; #2: $-x+1$, $y+1/2$, $-z$.

to L_2 *via* O3 for Sn1, O5 for Sn2; and are linked to benzoic anions *via* O1 for Sn1, O7#3 for Sn2. As shown in figure 5, the L_2 anions link Sn1 and Sn2 tricyclohexyltin groups to two polymeric chains. Face-to-face π–π interactions between the phenyl rings of adjacent L_2 anions are observed with a face-to-face distance of 3.612 Å (figure 6). The supramolecular architecture of **2** is a 2-D layer structure.

3.4.3. Molecular and crystal structures of 3. The structure of **3** contains one tricyclohexyltin cation and one L_3 anion (figure 7). The tin has distorted tetrahedral geometry with bond angles of 93.0(3)° to 123.2(6)°, the carboxylate oxygen of L_3 coordinates monodentate to tin giving a Sn1–O1 distance of 2.132(4) Å. The Sn–C bond lengths are in the range 2.144(5) to 2.150(7) Å, within the normal literature values [14].

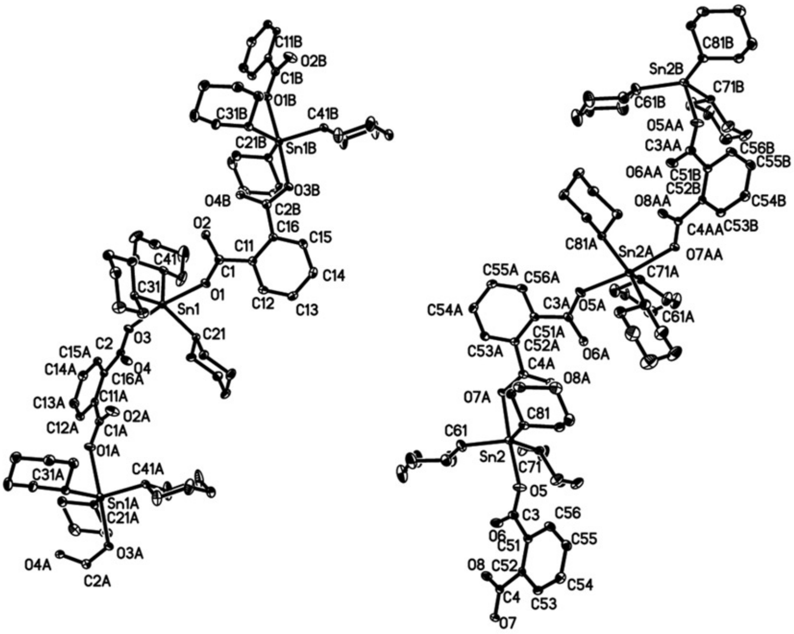


Figure 5. Repeating motif of the two independent structures of **2**.

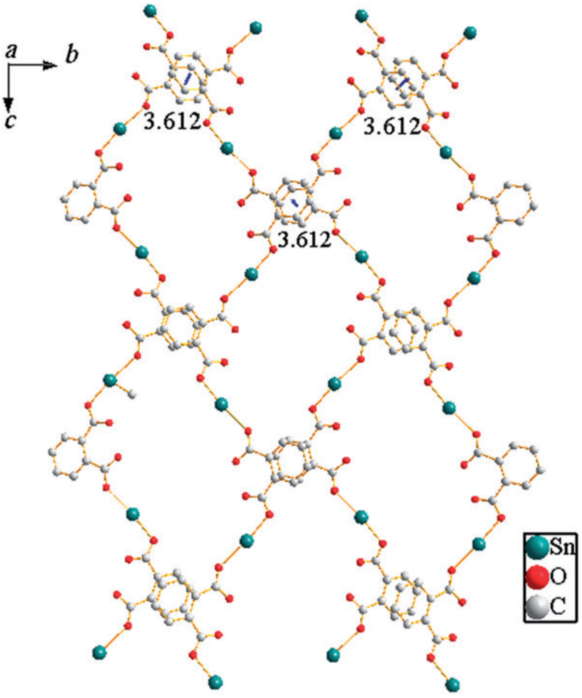


Figure 6. The 2-D supramolecular network of **2** formed by $\pi-\pi$ stacking interactions shown by blue dotted lines. Cyclohexyl groups have been omitted for clarity.

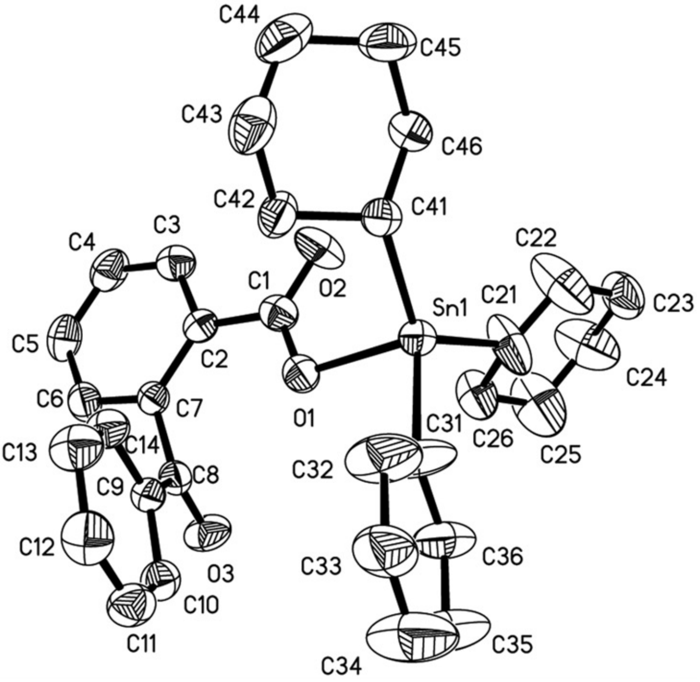


Figure 7. Molecule structure of **3** showing atomic numbering scheme. Hydrogen atoms have been omitted for clarity.

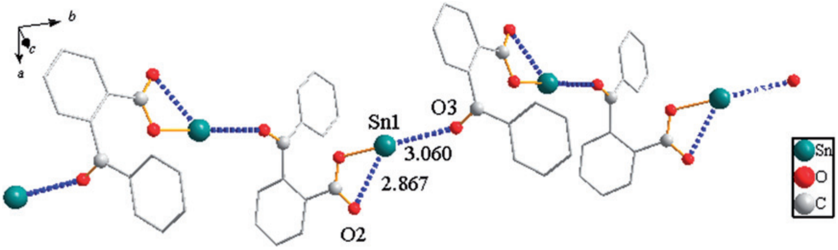


Figure 8. Polymeric chain of **3** formed by intramolecular weak O → Sn interactions.

The Sn ··· O interaction between O2 and Sn1 (at a distance of 2.867 Å) was smaller than the sum of Van der Waals radii 3.73 Å [15]. The structure of **3** is one of the two major motifs for $R'CO_2SnR_3$ [16]. Intermolecular Sn ··· O interactions exist between Sn1 and O3 at 3.060 Å. These Sn ··· O interactions lead the whole structure to a supramolecular chain, as shown in figure 8.

Supplementary material

CCDC-769210, 769211, and 769212 contain supplementary crystallographic data for **1**–**3**. 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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