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Crystal structures of two polymeric *bis*(triphenyltin) malonates

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In an attempt to prepare potassium/sodium salts of malonato-triphenylstannate, two *bis*(triphenyltin) malonates were obtained instead, $[(C_6H_5)_3Sn]_3[O_2CCH_2CO_2]_{1.5}$ (**1**) and $\{[(C_6H_5)_3SnO_2CCH_2CO_2Sn(C_6H_5)_3]H_2O\}CH_3CH_2OH$ (**2**). This provides an excellent example of structural diversity in triorganotin carboxylates. In both complexes, the dicarboxylate was connected to the triphenyltin groups forming a linear infinite polymeric chain. Both complexes have a *trans*-trigonal bipyramidal geometry with the three phenyl groups occupying the equatorial plane and two oxygens at axial positions. Both short and long tin–oxygen distances were observed in **1** and **2**. In **1**, all carboxylates functioned as bridging bidentate ligands, resulting in an infinite 3-D polymer network propagating along all three axes. In **2**, Sn1 is bonded to a carboxylate and a water molecule (Sn1–O1 2.145(3) Å, Sn1–O3 2.341(3) Å, O1–Sn1–O3 173.97(12)°). Only one carboxylate acts as a bidentate ligand allowing the carbonyl group to be axially coordinated to the adjacent tin, Sn2. Also, Sn2 is covalently bonded to the other carboxylate group in the malonate ligand (Sn2–O4 2.163(3) Å, Sn2–O5 2.353(3) Å, O4–Sn2–O5 173.47(12)°). There is a water molecule included in the crystal lattice hydrogen bonded to the uncoordinated carbonyl in malonate.

Keywords: X-ray crystallography; Triphenyltin; Malonates

1. Introduction

Organotin(IV) derivatives continue to attract attention due to their effectiveness as potential biocides [1–3] as well as structural considerations [4, 5]. Organotin complexes may interact with various biological systems as bactericides, fungicides, acaricides, and industrial biocides [1–3]. Furthermore, being effective antineoplastic agents (mainly antileukemic), they exhibit significant antitumor activity [6, 7]. Organotin carboxylates are well-known for their structural diversity. Triorganotin(IV) esters of monofunctional carboxylic acids can be either mono-, oligo-, poly-, or cyclooligomeric [4, 5]. In recent years, structural studies have focused on organotin dicarboxylates, as dicarboxylates are potential mono and multidentate ligands; they may also be intermolecularly

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bridging or intramolecularly chelating [8–16]. Self-assembly of organotin moieties and dicarboxylates can be applied to construct new organotin materials for a wide range of possible applications [8].

In order to increase activities for newly designed antitumor tin complexes, both solubility and lipophilicity of the molecule have been investigated [17, 18]. Aqueous solubility of organic molecules is an important molecular property that can affect biological activities; yet, a large number of pharmaceutically potential compounds including organotin compounds are only sparingly soluble [7]. Ongoing research in our laboratory has been directed toward the design and synthesis of ionic triorganotin complexes in the hopes of increasing their solubilities due to partial ionic characteristics. Previously, we reported the synthesis and crystal structure of an ammonium salt of thiolactatotriphenylstannate [19]. In an attempt to synthesize the potassium/sodium salts of malonato-triphenylstannate, the organic amine was replaced with an inorganic base (NaOH/KOH or NaHCO₃/KHCO₃). Two polymeric non-ionic triphenyltin complexes of malonic acid were obtained instead of the expected ionic triphenyltin complexes. While information on the structures of organotin dicarboxylates continues to accumulate, little has been reported on the structures of non-ionic triorganotin malonic acid [20]. In this article, we report the synthesis and X-ray crystal structures of two polymeric *bis*(triphenyltin) malonate complexes.

2. Experimental

2.1. Materials and measurements

IR spectra (400–4000 cm⁻¹) were recorded as KBr pellets on a Nicolet Magna-IR 760 spectrometer. All NMR measurements were made on a Varian Unity Inova 500 MHz spectrometer. Sample and instrument temperatures were at 298 K. Proton decoupled ¹³C- and ¹¹⁹Sn-spectra were acquired with a WALTZ decoupling sequence. ¹H- and ¹³C-chemical shifts were referenced to internal TMS while ¹¹⁹Sn-chemical shifts were referenced to tetramethyltin, externally.

2.2. Preparation

2.2.1. Synthesis of [(C₆H₅)₃Sn]₃[O₂CCH₂CO₂]_{1.5} (1). 0.521 g (5 mmol) of malonic acid was dissolved in 10 mL of water and 0.200 g (5 mmol) of NaOH was then added to the solution. A solution of triphenyltin hydroxide 1.835 g (5 mmol) was dissolved in 75 mL of absolute ethanol with heating and then added dropwise to the malonic acid solution with stirring. A white precipitate resulted and approximately 100 mL of absolute ethanol was added with heating. The solution was repeatedly stirred, heated, and filtered until it was clear. Slow evaporation at room temperature yielded crystals suitable for X-ray analysis. Yield 59%, m.p. 132°C. Analysis calculated for C_{22.5}H₁₈O₆Sn₃: C, 36.48; H, 2.43; Sn, 48.12%. Found: C, 36.73; H, 2.68; Sn, 47.87%.

2.2.2. Synthesis of {[(C₆H₅)₃SnO₂CCH₂CO₂Sn(C₆H₅)₃]H₂O}CH₃CH₂OH (2). 0.521 g (5 mmol) of malonic acid and 0.725 g (5 mmol) of K₂CO₃ were poured into 10 mL of

water and stirred until a clear solution was obtained. Ethanol was added dropwise until a cloudy solution appeared. Water was added dropwise to the cloudy solution until the solution became clear. A solution of triphenyltin chloride 1.926 g (5 mmol) was dissolved in 75 mL of absolute ethanol with heating. The triphenyltin chloride solution was then added dropwise to the malonic acid solution with stirring. A white precipitate resulted and approximately 100 mL of absolute ethanol was added with heating until the solution became clear. The solution was then filtered and slow evaporation at room temperature yielded suitable crystals for X-ray studies. Yield 39%, m.p. 164–166°C. Analysis calculated for $C_{41}H_{40}O_6Sn_2$: C, 56.85; H, 4.65; Sn, 27.41%. Found: C, 56.80; H, 4.55; Sn, 26.37%.

2.3. X-ray crystallography

Intensity data collections for **1** and **2** were carried out on a Bruker SMART Apex II X-ray diffractometer. Intensity data collection and cell determination protocols were carried out using graphite-monochromated Cu-K α radiation ($k = 0.71073 \text{ \AA}$). The data were corrected for Lorentz and polarization effects. Structure solution for each compound was obtained using direct methods (SHELXS-97) [21] and refined using the full-matrix least square methods on F^2 using SHELXL-97 [21]. Non-hydrogen atoms were refined with an anisotropic thermal parameter. The positions of hydrogens were either located in successive difference maps or were geometrically placed and refined using a riding model except for those hydrogens on distorted ethanol in **2**. Further details of the crystal data and refinement convergence are listed in table 1. Additional supporting information (CIF details for **1** and **2**) is available.

3. Results and discussion

3.1. Spectral studies

The mode of coordination of carboxylate to metals, including tin, has been deduced using the differences of the asymmetric and symmetric OCO vibrations [$\Delta\nu = \nu_{as}(\text{OCO}) - \nu_s(\text{OCO})$]. If $\Delta\nu$ is smaller than 250 cm^{-1} , then the carboxylate is considered bidentate [22, 23] forming a five-coordinate complex. Values less than 150 cm^{-1} have been assigned to chelated structures [22] while differences between 150 and 250 cm^{-1} have been assigned as bridged structures [22]. Differences greater than 250 cm^{-1} would indicate monodentate behavior for the ligand [23] and result in a four-coordinate compound. The observed differences for **1** and **2** are 182 and 171 cm^{-1} , respectively, indicating that in both complexes, the carboxylate is a bridged ligand in the solid state.

The $^1\text{H-NMR}$ spectra of both complexes show proton resonance due to triphenyltin and ligand moieties. Also, the number of protons calculated from the integrations of the resonances was in agreement with the calculated values expected for the molecules. In addition, a characteristic splitting pattern for ethanol was observed in **2**, indicating the existence of ethanol in the complex. ^{119}Sn -chemical shifts have been used to deduce the coordination number of tin in various organotin complexes [24]. The ^{119}Sn -chemical

Table 1. Crystallographic data and structure refinement parameters for **1** and **2**.

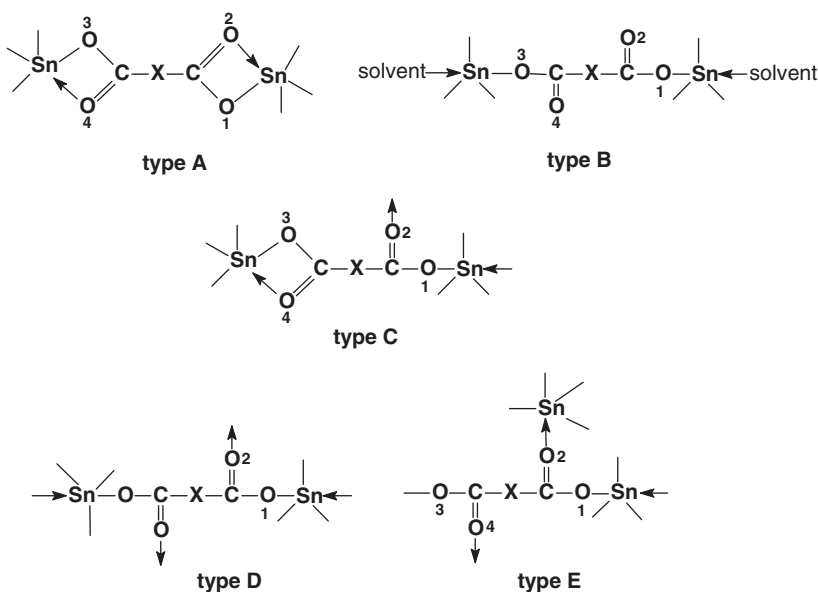
	Complex 1	Complex 2
Empirical formula	C _{58.50} H ₄₈ O ₆ Sn ₃	C _{39.50} H _{35.25} O ₆ Sn ₂
M_r	1203.04	843.31
Crystal system	Tetragonal	Tetragonal
Space group	$P4(3)2(1)2$	$I4$
Unit cell dimensions (Å, °)		
a	14.0340(2)	23.7013(2)
b	14.0340(2)	23.7013(2)
c	49.2861(9)	13.8973(3)
α	90	90
β	90	90
γ	90	90
Volume (Å ³), Z	9707.1(3), 8	7806.83(19), 8
$F(000)$	4776	3362
D_x (g mol ⁻¹ m ⁻³)	1.646	1.435
Absorption coefficient (mm ⁻¹)	12.60	10.51
Limiting indices	$h = -16 \leq 16;$ $k = -13 \leq 16;$ $l = -58 \leq 58$	$h = -27 \leq 28;$ $k = -27 \leq 27;$ $l = -13 \leq 16$
Max. and min. transmission	0.141 and 0.417	0.099 and 0.480
Measured reflections	106,190	39,959
Independent reflections	8669	6768
Reflections with $I > 2\sigma(I)$	8329	6665
R_{int}	0.056	0.034
θ range for data collection (°)	67.0°–3.6°	67.0°–3.7°
$R[F^2 > 2\sigma(F^2)]$	0.044	0.028
$wR(F^2)$	0.102	0.080
w	$1/[\sigma^2(F_o^2) + (0.0795P)^2]$	$1/[\sigma^2(F_o^2) + (0.0485P)^2 + 18.6903P]$
P	$(F_o^2 + 2F_c^2)/3$	$(F_o^2 + 2F_c^2)/3$
S	1.08	1.09
Parameters	610	435
$(\Delta/\sigma)_{max}$	0.003	0.003
Largest difference peak and hole (e Å ⁻³)	1.73 and –0.34	1.49 and –0.41

shifts of **1** and **2** in CDCl₃ solution showed a single sharp resonance at –101.7 and 105.8 ppm, respectively, similar to that reported for other four-coordinate triphenyltin carboxylates [25, 26].

Based on the IR and NMR results, both complexes are five-coordinate polymers in the solid state. However, in solution both complexes dissociate into four-coordinate monomers.

3.2. Crystal structures of **1** and **2**

Triorganotin esters of tetradentate dicarboxylates can be either monomeric or polymeric chains depending on the mode of coordination of the carboxylate, in which polymeric structures are formed through intermolecular Sn–O–C=O → Sn bonds [27]. Five different types of bonding modes have been reported for triorganotin dicarboxylates in the literature, as shown in scheme 1. Monomeric *bis*(triorganotin) dicarboxylates will be formed if the carbonyl group is prevented from approaching the tin in a neighboring molecule due to steric hindrance. The carboxylate in this bonding



Scheme 1. Possible bonding modes in triorganotin dicarboxylate.

type (A) is reported as chelating bidentate and the geometry around tin is *cis*-trigonal bipyramidal (*trans*-trigonal bipyramidal; TBP) [28]. Binuclear monomeric structure (type B) has also been reported for *bis*(triorganotin) dicarboxylate complexes when the coordination of the carbonyl groups is replaced by a solvent molecule, such as water in *bis*(triphenyltin) succinate [29].

When carboxylates are bridging bidentate, two types of polymeric structure are commonly reported [15, 16, 30]. Zigzag polymeric chains (type C) are obtained if the propagation of the polymer chain *via* one carboxylate is terminated. In this bonding mode, one carboxylate is chelating bidentate while the other is bridging bidentate [30]. The termination of carboxylate coordination has been reported as the result of steric hindrance of one carboxylate. In this case, one tin adopts a *cis*-TBP geometry while the other adopts a *trans*-TBP geometry with the three *R* groups in the equatorial plane and two oxygens in axial positions [30]. On the other hand, a macrocyclic structure is obtained when there is no steric hindrance or a solvent molecule that encumbers the coordination of both carboxylates [15, 16]. Bridging tetradentate dicarboxylate complexes are reported to have either a type D or E structure (scheme 1). In this case, both tins will adopt a *trans*-TBP geometry [15, 16].

The crystal data and structure refinements for $[(C_6H_5)_3Sn]_5[O_2CCH_2CO_2]_{1.5}$ (**1**) and $\{(C_6H_5)_3SnO_2CCH_2CO_2Sn(C_6H_5)_3\} \cdot CH_3CH_2OH$ (**2**) are listed in table 1. Additionally, selected bond lengths and angles for **1** and **2** are listed in tables 2 and 3. The molecular structures of **1** and **2** are shown in figures 1 and 2, respectively. The X-ray analyses indicate that both **1** and **2** are infinite polymeric *bis*(triphenyltin) malonates. The geometry around the tins in both complexes is essentially *trans*-TBP with the three phenyl groups occupying equatorial positions and two oxygens from the carboxylate at the axial position. An asymmetric bonding mode of the carboxylate

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

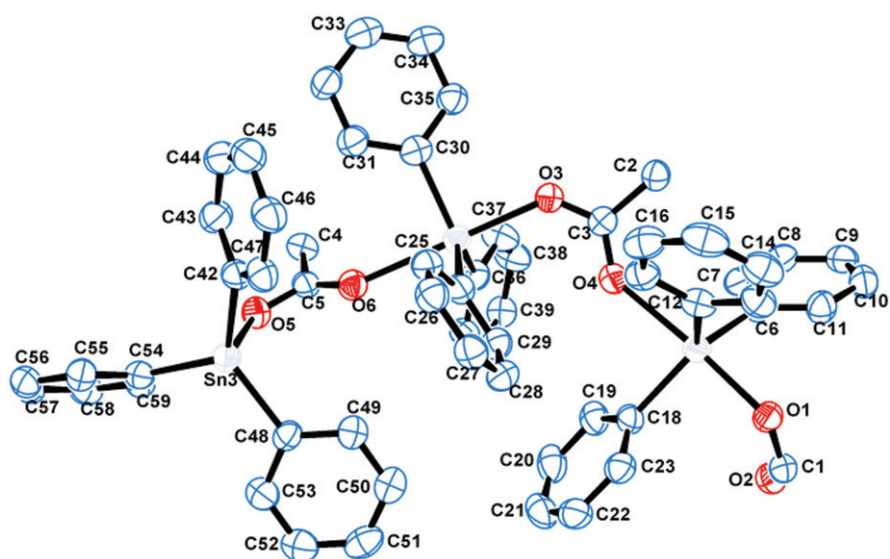
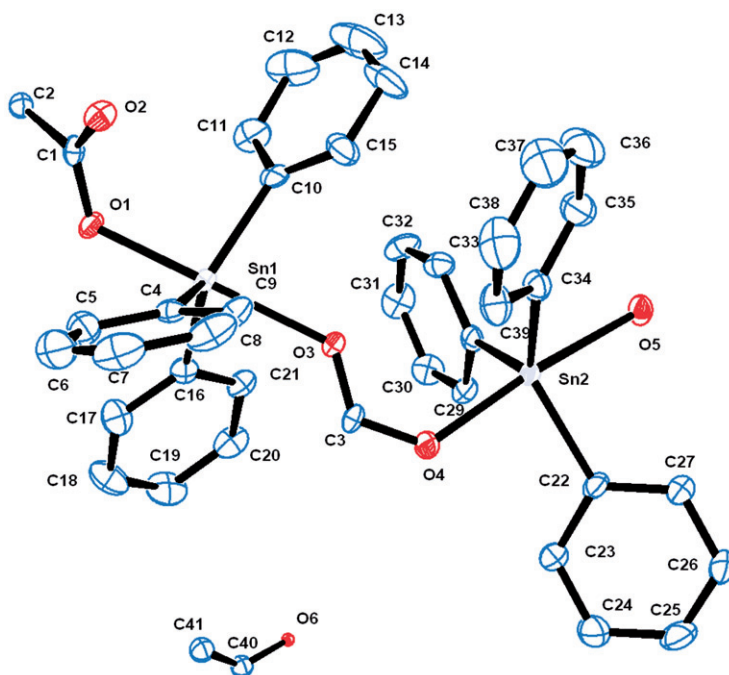
Sn1–C18	2.112(4)	Sn2–C36	2.104(4)	Sn3–C48	2.101(4)
Sn1–C6	2.117(4)	Sn2–C24	2.110(4)	Sn3–C42	2.109(4)
Sn1–C12	2.128(4)	Sn2–C30	2.115(4)	Sn3–C54	2.123(4)
Sn1–O1	2.172(3)	Sn2–O3	2.194(3)	Sn3–O5	2.159(3)
Sn1–O4	2.418(3)	Sn2–O6	2.480(3)	Sn3–O2	2.630(3)
O1–C1	1.293(5)	O3–C3	1.261(5)	O5–C5	1.280(5)
O2–C1	1.228(5)	O4–C3	1.260(5)	O6–C5	1.240(6)
C18–Sn1–C6	127.57(17)	C36–Sn2–C24	126.03(18)	C48–Sn3–C42	127.22(18)
C18–Sn1–C12	118.93(17)	C36–Sn2–C30	115.22(17)	C48–Sn3–C54	112.35(17)
C6–Sn1–C12	113.18(17)	C24–Sn2–C30	117.91(17)	C42–Sn3–C54	118.90(16)
C18–Sn1–O1	92.35(15)	C36–Sn2–O3	92.66(15)	C48–Sn3–O5	98.82(15)
C6–Sn1–O1	93.26(15)	C24–Sn2–O3	95.62(14)	C42–Sn3–O5	94.98(15)
C12–Sn1–O1	89.78(15)	C30–Sn2–O3	90.55(15)	C54–Sn3–O5	87.82(15)
C18–Sn1–O4	84.61(14)	C36–Sn2–O6	87.65(14)	C48–Sn3–O2	98.82(15)
C6–Sn1–O4	91.35(14)	C24–Sn2–O6	84.40(14)	C42–Sn3–O2	81.58(15)
C12–Sn1–O4	88.65(15)	C30–Sn2–O6	89.08(15)	C54–Sn3–O2	89.22(15)
O1–Sn1–O4	175.38(11)	O3–Sn2–O6	179.60(11)	O2–Sn3–O5	179.70(11)

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

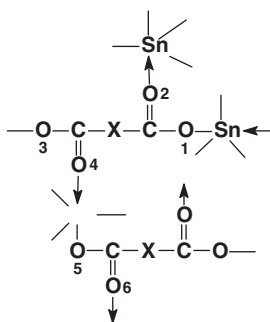
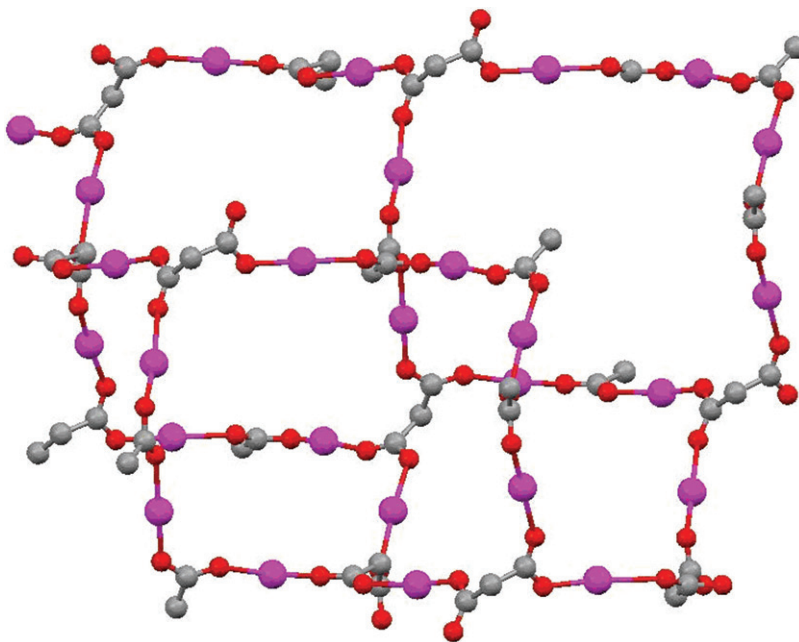
Sn1–C10	2.128(5)	Sn2–C28	2.127(5)
Sn1–C4	2.132(5)	Sn2–C34	2.136(5)
Sn1–C16	2.134(4)	Sn2–C22	2.140(5)
Sn1–O1	2.145(3)	Sn2–O4	2.163(3)
Sn1–O3	2.341(3)	Sn2–O5	2.353(3)
O1–C1	1.303(6)	O3–C3	1.241(6)
O2–C1	1.233(6)	O4–C3	1.276(5)
C10–Sn1–C4	122.25(19)	C28–Sn2–C34	119.0(2)
C10–Sn1–C16	119.81(18)	C28–Sn2–C22	118.65(19)
C4–Sn1–C16	116.97(17)	C34–Sn2–C22	121.7(2)
C10–Sn1–O1	93.70(16)	C28–Sn2–O4	95.40(15)
C4–Sn1–O1	96.80(16)	C34–Sn2–O4	96.99(15)
C16–Sn1–O1	89.18(16)	C22–Sn2–O4	85.49(15)
C10–Sn1–O3	85.14(15)	C28–Sn2–O5	83.37(15)
C4–Sn1–O3	88.80(15)	C34–Sn2–O5	89.19(15)
C16–Sn1–O3	86.29(15)	C22–Sn2–O5	89.47(15)
O1–Sn1–O3	173.97(12)	O4–Sn2–O5	173.47(12)

oxygen is observed as both short and long tin–oxygen distances are observed in the complexes.

In **1**, all the carboxylates function as bridging bidentate ligands, allowing propagation of the carboxylate along all three axes to give a 3-D polymer network. Bonding for the complex is a combination of type D and E (scheme 2). Eight tins and eight malonates are linked to form a 36-membered macrocyclic ring (figure 3). This bonding pattern makes enough space in the lattice to include ethanol molecules in the unit cell, as shown in the crystal structure. All the tins are of *trans*-TBP geometry, and both short and long tin–oxygen distances are observed for all three tins. The longer tin–oxygen bonds within the pair of values for Sn1 (2.172(3), 2.418(3) Å), Sn2 (2.194(3), 2.480(3) Å), and Sn3 (2.159(3), 2.630(3) Å) are clearly of the dative type.

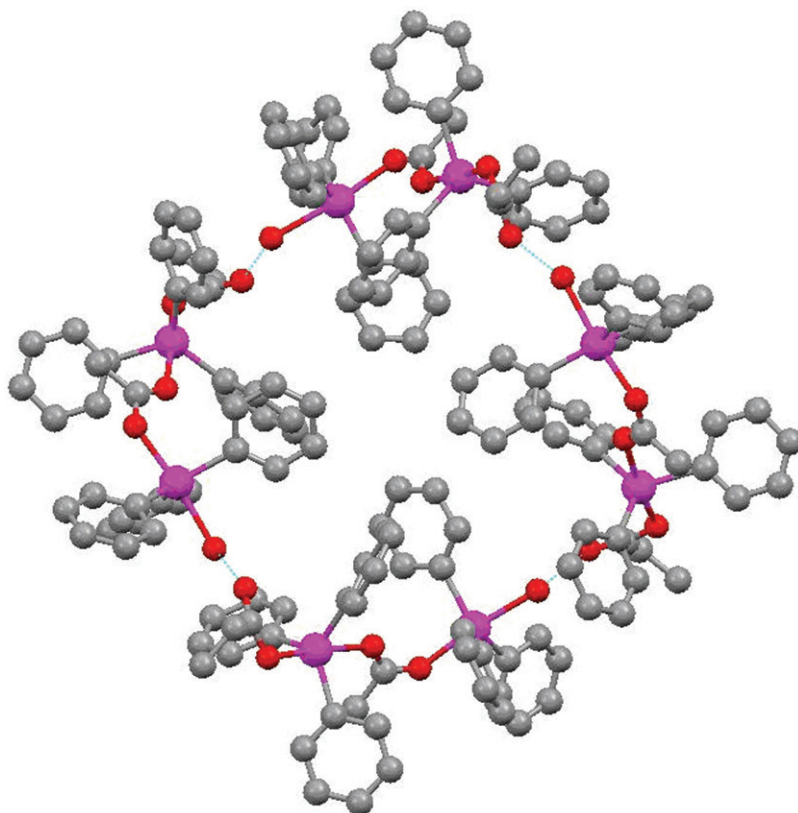
Figure 1. Repeating unit of **1**.Figure 2. Repeating unit of **2**.

In **2**, the coordination competition from a water causes termination of one carboxylate coordination to tin. Only one carboxylate is bidentate while the other carboxylate is monodentate. The polymeric structure formed is very similar to type C depicted in scheme 1. The Sn1 is covalently bonded to monodentate carboxylate O1.

Scheme 2. Polymeric bonding mode in **1**.Figure 3. Polymeric 3-D network in **1** (phenyl groups omitted for clarity).

The other axial position is occupied by the carbonyl oxygen (O3) of the bidentate carboxylate (Sn1–O1 2.145(3) Å, Sn1–O3 2.341(3) Å, O1–Sn1–O3 173.97(12)°). The O4 from the bidentate carboxylate is covalently bonded to Sn2 and O5 from a water molecule is the second axial O atom (Sn2–O4 2.163(3) Å, Sn2–O5 2.353(3) Å, O4–Sn2–O5 173.47(12)°), resulting in the formation of a polymeric chain.

The existence of a water molecule in the lattice stabilizes the crystal structure through hydrogen bonding between oxygen of water and the uncoordinated carbonyl in malonate (figure 4). Hydrogen bonding (table 4) (O5–H1W...O2^{vi}, symmetry codes: (vi) $-y + 1, x + 1, -z + 2$) substantially affects distribution of the electrons on the non-coordinating carbonyl, which can be seen by the observation of a C=O length of 1.303(6) Å for the C(1)–(O(2)) bond which is longer than the normal C=O length.

Figure 4. Hydrogen bonding in **2**.Table 4. Hydrogen-bond parameters (\AA , $^\circ$) for **2**.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O5-H1W \cdots O2^{vi}$	0.85	1.91	$2.660^\circ(5)$	147

Symmetry codes: $vi-y+1, x+1, -z+2$.

4. Summary

While the desired ionic triorganotin complexes were not obtained, two triorganotin carboxylates were synthesized instead. The bonding of the bridging tetradentate dicarboxylate in **1** is a combination of two bonding structures reported in other similar bridging tetradentate dicarboxylate complexes. The second complex (**2**) contains carboxylates which function as both monodentate and bidentate. Bonding and structures of the triorganotin dicarboxylates in these complexes are not typical of those observed in similar triorganotin dicarboxylates.

Supplementary material

CCDC-704322 and CCDC-704321 contains crystallographic data for **1** and **2**. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Center (CCDC), 12 Union Road, Cambridge CB2 1 EZ, UK; Fax: +44(0)1223-336033; E-mail: deposit@ccdc.cam.ac.uk).

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