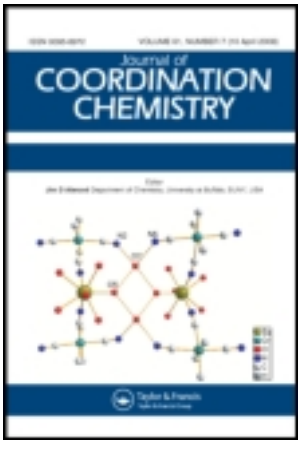

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Synthesis and characterization of polymeric triphenyltin and cyclotetrameric tricyclohexyltin 2-(1H-imidazol-1-yl)acetates

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Synthesis and characterization of polymeric triphenyltin and cyclotetrameric tricyclohexyltin 2-(1*H*-imidazol-1-yl)acetates

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The reaction of 2-(1*H*-imidazol-1-yl)acetic acid with $(Ph_3Sn)_2O$ or Cy_3SnOH (Cy = cyclohexyl) yields triphenyltin 2-(1*H*-imidazol-1-yl)acetate (**1**) and tricyclohexyltin 2-(1*H*-imidazol-1-yl)acetate (**2**), respectively. 2-(1*H*-imidazol-1-yl)acetates in these two complexes show remarkably different coordination modes. Complex **1** forms a polymeric chain structure through intermolecular Sn–N interactions, while **2** displays a 28-membered macrocyclic tetranuclear structure by the assembly of Sn–N coordination bonds.

Keywords: Organotin carboxylate; 2-(1*H*-imidazol-1-yl)acetic acid; X-ray crystal structure

1. Introduction

Although industrial and agricultural applications of organotin compounds are partially limited because of the toxicity and environmental effects, the synthesis and characterization of organotin carboxylates continue to be an active research area owing to their remarkable structural diversity [1–3] as well as significant biological activities [4–8] such as pesticidal, antibacterial, and antitumor agents. Recently, cyclooligomeric organotin carboxylates have attracted attention owing to their fascinating structures and potential applications. Multifunctional carboxylic acids with additional oxygen [9–11], sulfur [12–14], or nitrogen [15–18] donors and polycarboxylic acids [18–22] are usually used for the construction of cyclooligomeric structures [23]. Many macrocyclic di- [11], tri- [24, 25], tetra- [20, 21], and hexanuclear [10, 13, 15] diorganotin carboxylates have been synthesized by this strategy. Some macrocyclic di- [26], tetra- [24, 27–30], and hexanuclear [12, 19, 22, 31, 32] triorganotin carboxylates have also been reported. As a flexible multifunctional ligand, 2-(1*H*-imidazol-1-yl)acetic acid exhibits variable coordination modes in the construction of coordination architectures by the use of carboxylate oxygens and the exodentate nitrogens on imidazole. Some transition-metal complexes with interesting structural frameworks containing this ligand have been described [33–35]. Lately, we have been interested in the synthesis of organotin

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carboxylates with additional nitrogen donors, which exhibit considerable structural diversity and good antifungal activities [17, 36, 37]. For example, triphenyltin 1*H*-tetrazolyl-1-acetate displays a cyclohexameric structure by the assembly of a Sn–N coordination bond instead of the common Sn–O bond, while tricyclohexyltin 1*H*-tetrazolyl-1-acetate forms a polymeric chain structure through intermolecular Sn···N interactions [17]. Taking into consideration some similar structural features of 2-(1*H*-imidazol-1-yl)acetic acid with 1*H*-tetrazolyl-1-acetic acid, such as having exodentate nitrogen as well as the carboxylate, we investigated the reaction of 2-(1*H*-imidazol-1-yl)acetic acid with $(Ph_3Sn)_2O$ and Cy_3SnOH (Cy = cyclohexyl) and found that the structures of triphenyltin 2-(1*H*-imidazol-1-yl)acetate (**1**) and tricyclohexyltin 2-(1 *H*-imidazol-1-yl)acetate (**2**) are remarkably different from those of the corresponding analogues of 1 *H*-tetrazolyl-1-acetate.

2. Experimental

2.1. *Materials and measurements*

NMR spectra were obtained with a Bruker 400 spectrometer using DMSO-d_6 as solvent, and the chemical shifts were reported in parts per million with respect to reference standards (internal $SiMe_4$ for ^{1}H- and ^{13}C-NMR, and external Me_4Sn for ^{119}Sn-NMR (149 MHz) spectra). IR spectra were obtained from a Shimadzu FTIR 8400S as KBr discs. Elemental analyses were carried out on an Elementar Vairo EL analyzer. Melting points were measured with an X-4 digital micro melting-point apparatus and were uncorrected. All chemicals are commercially available and were used as received without purification.

2.2. *Synthesis of 1*

Mixture of 2-(1*H*-imidazol-1-yl)acetic acid (0.13 g, 1 mmol) and $(Ph_3Sn)_2O$ (0.36 g, 0.5 mmol) in anhydrous benzene (30 mL) and methanol (30 mL) was stirred and heated at reflux for 24 h. After cooling to room temperature, a white precipitated solid was filtered off and recrystallized from methanol to yield colorless crystals of **1**. Yield: 0.43 g (90%); m.p. 247–249°C. ^{1}H-NMR: δ 7.83–7.67 (m, 6H, C_6H_5), 7.45–7.39 (m, 9H, C_6H_5), 7.36, 6.91, 6.78 (s, s, s, 1H, 1H, 1H, protons of imidazole), 4.56 (s, 2H, CH_2) ppm. Anal. Calcd for $C_{23}H_{20}N_2O_2Sn$ (%): C, 58.14; H, 4.24; N, 5.90. Found (%): C, 58.25; H, 4.21; N, 5.85. IR (cm^{-1}): 3147, 3066, 3046, 1665 [ν_{as}(COO)], 1585, 1520, 1481, 1429, 1367 [ν_s(COO)], 1294, 1272, 1234, 1088, 997, 941, 827, 785, 739, 695, 689, 653.

2.3. *Synthesis of 2*

This complex was obtained similarly using tricyclohexyltin hydroxide instead of $(Ph_3Sn)_2O$ as described above for **1**, but in a 1 : 1 molar ratio. After heating at reflux for 10 h, the solvent was removed under reduced pressure to give a white solid, which was recrystallized from benzene to yield colorless crystals of **2**. Yield: 85%; m.p. 160–162°C. ^{1}H-NMR: 7.52, 7.03, 6.84 (s, s, s, 1H, 1H, 1H, protons of imidazole), 4.62 (s, 2H, CH_2),

1.83–1.80, 1.64–1.56, 1.23–1.14 (m, m, m, 9H, 18H, 6H, C_6H_{11}) ppm. Anal. Calcd for $C_{23}H_{38}N_2O_2Sn$ (%): C, 56.00; H, 7.76; N, 5.68. Found (%): C, 55.85; H, 7.97; N, 5.67. IR (cm^{-1}): 2921, 2847, 1645 [ν_{as}(COO)], 1513, 1446, 1373 [ν_s(COO)], 1238, 1172, 1082, 994, 927, 731, 693, 662.

2.4. *Crystal structure determination*

Crystals of **1** suitable for X-ray analysis were obtained by slowly cooling hot methanol/benzene solution; crystals of **2** suitable for X-ray analysis were obtained by slow diffusion of hexane into CH_2Cl_2 solution at room temperature. All intensity data were collected with a Rigaku Saturn724 CCD diffractometer using graphite-monochromated Mo-Kα radiation ($\lambda = 0.71075$ Å) at 113(2) K. Semi-empirical absorption corrections were applied and all calculations were performed using the Crystalclear program [38]. The structures were solved by direct methods and difference Fourier maps using SHELXS of the SHELXTL package and refined with SHELXL [39] by full-matrix least-squares on F^2. The cyclohexyl group (C18–C23) in **2** was disordered and the site occupation factor of these disordered atoms was adjusted (0.50 for C18–C23) to give reasonable thermal parameters. All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogens were added geometrically and refined with riding model position parameters. A summary of the fundamental crystal data for **1** and **2** is listed in table 1.

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

Complex	**1**.0.5C_6H_6	**2**.0.5CH_2Cl_2
Formula	$C_{26}H_{23}N_2O_2Sn$	$C_{23.5}H_{39}ClN_2O_2Sn$
Formula weight	514.15	535.71
Crystal system	Monoclinic	Triclinic
Space group	*C*2/*c*	$P\bar{1}$
Unit cell dimensions (Å, °)		
a	20.755(4)	12.6140(10)
b	14.6380(15)	12.6280(11)
c	16.9000(17)	16.5340(16)
α	90	99.457(5)
β	116.195(6)	93.7760(10)
γ	90	107.396(5)
Volume (Å^3), *Z*	4607.1(11), 8	2460.5(4), 1
Calculated density (g cm^{-3})	1.483	1.446
Absorption coefficient (mm^{-1})	1.133	1.168
F(000)	2072	1108
Crystal size (mm)	0.26 × 0.22 × 0.14	0.22 × 0.18 × 0.16
2θ range for data collection (°)	3.54–55.72	2.52–55.76
Reflections collected	23,096	31,619
Independent reflections	5492 [R(int) = 0.0393]	11,691 [R(int) = 0.0375]
Observed reflections [$I > 2\sigma(I)$]	4717	8855
Parameters	280	587
Goodness-of-fit on F^2	1.036	0.973
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0287$, $wR_2 = 0.0723$	$R_1 = 0.0285$, $wR_2 = 0.0710$

3. Results and discussion

3.1. *Synthesis and characterization of complexes*

Compounds **1** and **2** have been synthesized by the reaction of 2-(1*H*-imidazol-1-yl)acetic acid with $(Ph_3Sn)_2O$ or Cy_3SnOH (Cy = cyclohexyl), respectively. These complexes have been characterized by IR and ^{1}H-NMR spectra as well as elemental analyses.

In IR spectra the difference between asymmetric and symmetric carboxylate stretching vibrations has been widely used to deduce the coordination mode of carboxylate in the solid state [40]. Generally, when the difference is larger than the corresponding value in ionic compounds, a monodentate coordination mode is suggested. IR spectra of **1** and **2** display remarkable differences between asymmetric and symmetric stretching vibrations of the carboxylate (298 cm^{-1} in **1** and 272 cm^{-1} in **2**). These values are markedly larger than that observed in the corresponding sodium salt of 2-(1*H*-imidazol-1-yl)acetic acid (217 cm^{-1}), in which the asymmetric and symmetric stretching vibrations of the carboxylate are observed at 1615 and 1398 cm^{-1}, respectively. This result implies that the carboxylates in **1** and **2** are monodentate [40]. The ^{1}H-NMR spectroscopic data are consistent with the suggested structures, exhibiting the expected integration values and chemical shifts. Due to the low solubility of these two complexes, their satisfactory ^{119}Sn- and ^{13}C-NMR signals were not observed.

3.2. *The crystal structures of 1 and 2*

To verify the role of the heteroatoms, the structures of **1** and **2** have also been confirmed by X-ray crystallography. The molecular structure of **1** (figure 1) forms a linkage coordination polymer through intermolecular Sn–N interactions, similar with tri(p-tolyl)tin 3-(4*H*-1,2,4-triazol-4-yl)benzoate (**A**) [37], but remarkably different from the cyclohexameric structure of triphenyltin 1*H*-tetrazolyl-1-acetate (**B**) [17]. The 2-(1*H*-imidazol-1-yl)acetate is a bridging bidentate ligand by the carboxylate oxygen and exodentate nitrogen of imidazole. The carboxylate coordinates to tin monodentate, consistent with the result of its IR spectrum. The tin is a five-coordinate distorted trigonal bipyramid with the electronegative nitrogen and oxygen occupying the axial positions. The axial N2–Sn1–O2A angle is 177.59(6)°, slightly deviating from linearity. The Sn1–N2 bond distance is 2.353(2) Å in **1**, shorter than those in other reported triaryltin derivatives with azolyl-functionalized carboxylate ligands, such as **A** (2.4509(18) Å) and **B** (2.609(6) Å), possibly due to the stronger donor ability of nitrogen of imidazole compared to the corresponding nitrogens of triazole and tetrazole. The non-bonded Sn1···O1A distance is 3.199(2) Å, shorter than the sum of the van der Waals radii for Sn and O [23], indicating the presence of weak interactions. In addition, some weak intermolecular C–H···O hydrogen-bonding interactions have been observed in the crystal packing of **1** (figure 2), such as C2–H2···O1^i (H2···O1^i/C2···O1^i distances: 2.480(2)/3.263(3) Å; symmetry operation i: $0.5-x$, $0.5-y$, $-z$). This complex forms a 2-D supramolecular structure through these weak intermolecular C–H···O hydrogen-bonding interactions.

Although some macrocyclic tetrameric triorganotin carboxylates have been described in the literature [24, 27–30], they are generally formed by the assembly of the Sn–O bond. Furthermore, cyclooligomeric structures for tricyclohexyltin carboxylates are

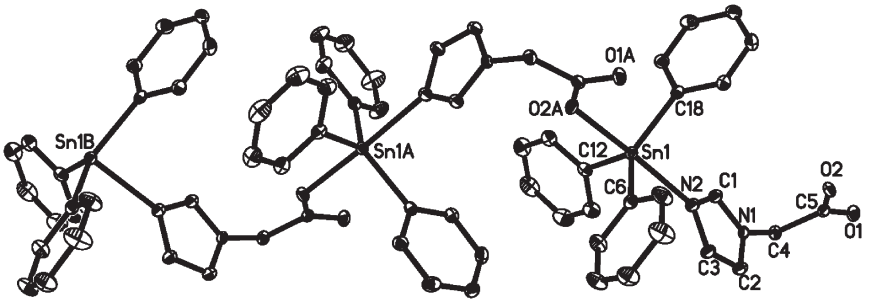

Figure 1. The polymeric chain structure of **1** with thermal ellipsoids at the 30% probability level. The uncoordinated solvents and hydrogens are omitted for clarity. Selected bond distances (Å) and angles (°): Sn1–N2, 2.353(2); Sn1–O2A, 2.175(2); Sn1 ··· O1A, 3.199(2); C5–O1, 1.230(3); C5–O2, 1.283(3); C4–N1, 1.456(3) Å; and N2–Sn1–O2A, 177.59(6)°; C6–Sn1–N2, 85.79(7)°; C6–Sn1–C18, 130.33(8)°; C12–Sn1–O2A, 87.92(7)°; O1–C5–O2, 126.3(2)°. Symmetric operations: A = $-x+0.5$, $y-0.5$, $-z+0.5$; B = x, $y-1$, z.

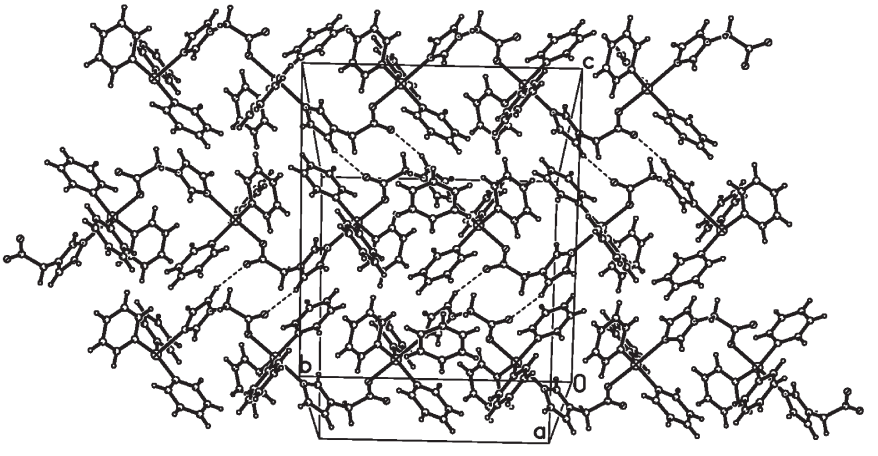

Figure 2. The crystal packing diagram of **1** emphasizing the weak intermolecular interactions (H2 ··· O1^{i}/C2 ··· O1^{i} distances: 2.480(2)/3.263(3) Å; symmetry operation: i = $0.5-x$, $0.5-y$, $-z$) and the 2-D network.

unknown. To our knowledge, the formation of the cyclotetrameric structure of triorganotin carboxylates through Sn–N bonds is not reported. Compound **2** (figure 3) exhibits a 28-membered macrocyclic tetrameric structure through intermolecular Sn–N interactions, instead of a linkage coordination polymer as in **1** as well as tricyclohexyltin 1*H*-tetrazolyl-1-acetate (**C**) [17]. The Sn1–N1 distance of 2.499(2) Å is markedly shorter than that in the corresponding complex of 1*H*-tetrazolyl-1-acetate (**C**) (2.924(9) Å) [17], indicating relatively stronger Sn–N interactions in **2** than in **C**. The tins in **2** also adopt a five-coordinate distorted trigonal bipyramidal geometry like **1**. The axial N–Sn–O angle is 177.52(7)°, very close to that in **1**. In addition, some weak interactions between the Sn1 and O4 as well as Sn2 and O2 exist, verified by the non-bonded Sn1 ··· O4 distance (3.270(2) Å) and Sn2 ··· O2 distance (3.370(2) Å), which are shorter than the sum of the van der Waals radii for Sn and O [23].

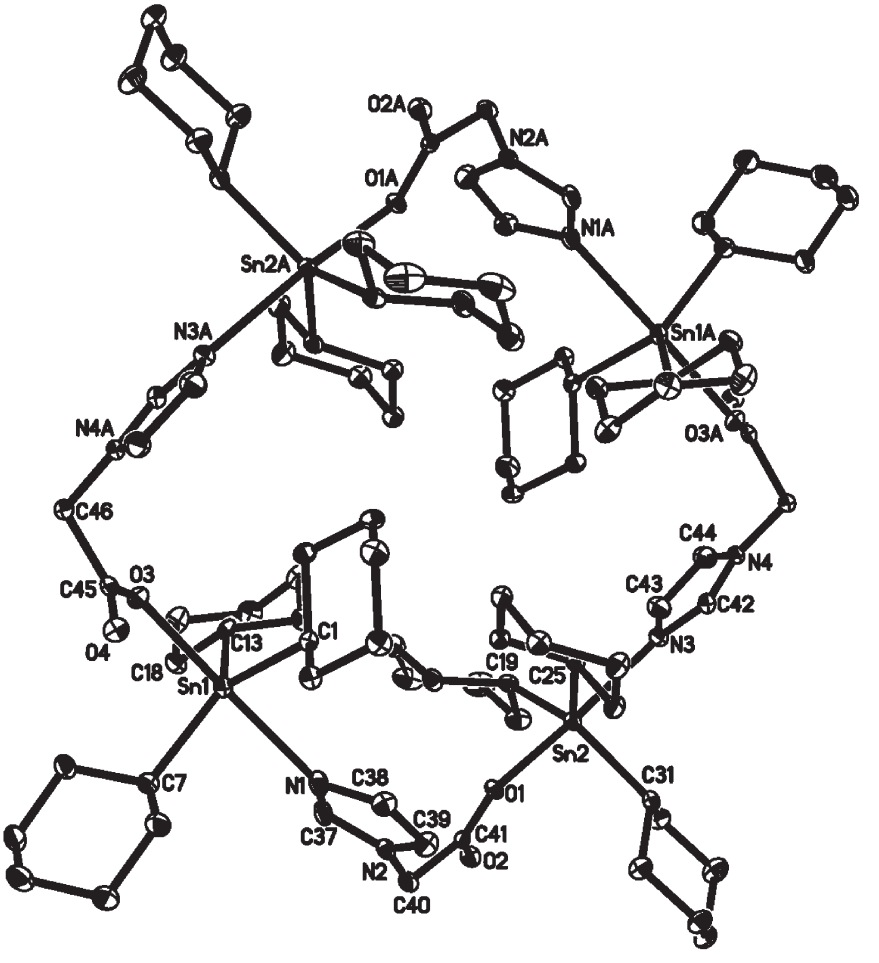

Figure 3. The cyclotetrameric structure of **2** with thermal ellipsoids at the 30% probability level. The uncoordinated solvents and hydrogens are omitted for clarity. Selected bond distances (Å) and angles (°): Sn1–N1, 2.499(2); Sn1–O3, 2.214(2); Sn1 ··· O4, 3.270(2); C41–O1, 1.282(3); C45–O3, 1.274(4); Sn2–N3, 2.508(2); Sn2–O1, 2.202(2); Sn2 ··· O2, 3.370(2); C41–O2, 1.227(3); C45–O4, 1.240(3) Å; and O1–C41–O2, 127.3(2)°; O3–C45–O4, 126.4(3)°; N2–C40–C41, 115.6(2)°; N4A–C46–C45, 109.1(2)°; N1–Sn1–O3, 177.52(7)°; C7–Sn1–O3, 97.06(8)°; C1–Sn1–C7, 127.70(9)°; C7–Sn1–N1, 84.60(8)°; C1–Sn1–C13, 110.6(2)°; O1–Sn2–N3, 173.46(6)°; C31–Sn2–O1, 96.48(8)°; C19–Sn2–C25, 118.38(9)°; C19–Sn2–N3, 84.51(8)°; C25–Sn2–C31, 116.69(9)°. Symmetric operations: A = $-x+1$, $-y+1$, $-z$.

A series of weak intermolecular C–H ··· O and C–H ··· Cl hydrogen bonds also exist in the crystal packing of **2**. The C37–H37 ··· O2^{ii} and C47–H47B ··· O4^{iii} distances (symmetry operations ii: $-x$, $-y$, $-z$ and iii: $1-x$, $1-y$, $1-z$) are 2.272(2)/3.168(3) nm (H37 ··· O2^{ii}/C37 ··· O2^{ii}) and 2.337(2)/3.202(5) Å (H47B ··· O4^{iii}/C47 ··· O4^{iii}). The C17–H17B ··· Cl2 distance is 2.876(1)/3.587(7) Å for H17B ··· Cl2 and C17 ··· Cl2, respectively. These weak interactions play important roles in stabilizing the crystal framework.

In conclusion, compounds **1** and **2** have been synthesized by the reaction of 2-(1*H*-imidazol-1-yl)acetic acid with $(Ph_3Sn)_2O$ or Cy_3SnOH (Cy = cyclohexyl), respectively.

The former forms a linkage coordination polymer while the latter displays a rare cyclotetrameric structure for tricyclohexyltin carboxylates through intermolecular Sn–N interactions. The structural features of these two complexes are markedly different from those of the corresponding organotin 1*H*-tetrazolyl-1-acetates.

Supplementary material

CCDC-807963 for **1** and 807964 for **2** contain the supplementary crystallographic data for this article. These data can be obtained free of charge from CCDC, 12 Union Road, Cambridge, CB21EZ, UK (Fax: +44(0)1223-336033; Email: deposit@ccdc.cam.ac.uk).

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