ORIGINAL ARTICLE

A C G publications

Org. Commun. 5:1 (2012) 18-26

organic communications

Organotin(IV) compounds of *n*-propyl and isopropyl glutarate: Their synthesis, spectral characterization and antibacterial activity

Javid Hussain[1,2], Hidayat Hussain[2*], Viqar Uddin Ahmad[3], Ahmed Al-Harrasi[2] and Amin Badshah[4]

[1]*Department of Chemistry, Kohat University of Science and Technology Kohat, Khyber Pakhtunkhwa, Pakistan*

[2]*Department of Biological Sciences and Chemistry, PC 616, University of Nizwa, Nizwa, Sultanate of Oman*

[3]*H.E.J. Research Institute of Chemistry, International Center for Chemical Sciences, University of Karachi, Karachi 75270, Pakistan*

[4]*Department of Chemistry, Quaid-e-Azam University, Islamabad, Pakistan*

(Received October 20, 2011; Revised February 12, 2012; Accepted February 20, 2012)

Abstract: A series of new di- and triorganotin(IV) complexes of monoisopropyl and mono *n*-propyl glutarate have been synthesized and characterized by ^{1}H- ^{13}C- ^{119}Sn-NMR, UV, IR and Mass spectrometry. The spectroscopic investigation demonstrated that the carboxylate group acts as a bidentate ligand in diorganotin(IV) and as a monodentate ligand in triorganotin(IV) compounds. Biological evaluation against various microorganisms indicates that the diorganotin(IV) complexes are slightly less reactive than triorganotin(IV) complexes.

Keywords: Organotin(IV) carboxylates; spectroscopic studies; biological activity.

1. Introduction

Organotin(IV) compounds of the carboxylic acids are being extensively studied with special reference to their methods of synthesis, structural elucidation and biological activity [1,2]. Organotin compounds show a large spectrum of biological properties such as antitumor, antifungal, and antibacterial [1,2].

As a continuation of our series on the synthesis and characterization of organotin carboxylates [1], we report here the synthesis and spectral characterization of organotin(IV) derivatives of *n*-propyl and isopropyl glutarate. Different carboxylate ligands were prepared by treating maleic anhydride and glutaric anhydride with different alcohols.

* Corresponding author: *E-Mail:*Hidayat110@gmail.com

The article was published by Academy of Chemistry of Globe Publications
www.acgpubs.org/OC/index.htm © Published 03/30/2012 EISSN:1307-6175

2. Results and discussion

Two series of complexes i.e diorganotin(IV) (1-5) and triorganotin(IV) (6-8) (Figure 1; Scheme 1) were subsequently prepared by reactions of monoester ligand (HL) and different organotin halides in 1:1 and 2:1 (ligand : metal) molar ratios and is illustrated in Scheme 1. The reactions of dibutyltin(IV) oxide with carboxylate ligands in a toluene-ethanol (3:1, v/v) mixture afforded the complexes in a 1:2 molar ratio with the azeotropical removal of water. All the complexes are almost soluble in every organic solvent at room temperature.

$$R_2SnCl_2 + 2HL + 2Et_3N \longrightarrow R_2SnL_2 + 2Et_3N.HCl \quad (1)$$

R = methyl and pheny

HL = monoisopropyl and *n*-propyl glutarate

$$n\text{-}Bu_2SnO + 2HL \longrightarrow n\text{-}Bu_2SnL_2 + H_2O \quad (2)$$

HL = mono *n*-propyl glutarate

$$RSnCl_3 + 3LHEt_3N \longrightarrow C_6H_5SnL_3 + 3Et_3N.HCl \quad (3)$$

R = methyl and pheny

HL = monoisopropyl and *n*-propyl glutarate

Scheme 1. Prepared the two series of complexes i.e diorganotin(IV) (1-5) and triorganotin(IV)

2.1.Electronic Spectra

The spectra of the ligands and their complexes were recorded in absolute ethanol. The various bands observed were assigned to interligand and charge transfer or $n\rightarrow\pi^*$ transitions according to their energies and intensities. It was found that the electronic spectra of these complexes exhibit very intense bands in the range of 200-210 nm, which may be due to the $n\rightarrow\pi^*$ transition of the (COO) chromophore [2,3]. Furthermore, there is a sharp band observed in the 255-270 nm region in the spectra of the complexes, which is ascribed to the charge-transfer band since it is known that metal/metalloids are capable of forming $d\pi$-$p\pi$ bands with ligands containing nitrogen or oxygen as donor atoms [2,3]. The tin atom has vacant 5d orbitals and hence $L\rightarrow M$ electronic orbital overlap can take place by the acceptance of a pair of electrons from an oxygen donor atom of the ligand [2,3].

2.2. Infrared Spectroscopy

The IR spectra of these compounds have been recorded in the range of 400-4000 cm^{-1}. Tentative assignments have been made on the basis of earlier publications and the important data are listed in Table 1. The absorptions of interest in the spectra of the complexes are ν(COO), ν(Sn-C) and ν(Sn-O). The absence of the ν(OH) in all the organotin (IV) compounds in the 2500-3000 cm^{-1} region, and the presence of ν(Sn-O) in the 450-475 cm^{-1} range [2,4] indicates deprotonation of the carboxylic acid group and consequent coordination of the carboxylate group with the tin metal as expected. The vacant 5d orbital of tin atoms tends toward high coordination with ligands containing lone pairs of electrons. The IR stretching frequencies of the carboxylate groups are very important for determining their structures viz., when there are interactions between the carbonyl oxygen atoms of the carboxylate groups and the tin atom, the asymmetric absorption vibration frequencies ν_{asym} (CO_2) of the carboxylate groups decrease and the symmetric absorption frequencies ν_{sym} (CO_2) increase. Their difference viz., $\nu\Delta$ (CO_2), therefore decreases [3]. In the IR spectra of the title compounds the carboxylate bands are observed in the characteristic regions for ν_{asym} (CO_2) between 1640 and 1575

cm^{-1} and for ν_{sym} (CO_2) between 1420 and 1310 cm^{-1} (Table 1). In the triorganotin (IV) derivatives, **6-8**, the differences [$\nu\Delta$ (CO_2)] between ν_{asym} (CO_2) and ν_{sym} (CO_2) are more than 200 cm^{-1} indicating the covalent nature of the metal-oxygen bond [2,5]. Ionic bonding and also bridging or chelation, therefore, can be excluded, and unidentate coordination of the carboxylic groups bonding to the metal must therefore be assumed [2,4]. In the diorganotin (IV) derivatives **1–5**, the frequencies ν_{sym} (CO_2) are shifted to higher wave numbers compared to those of the free acids.

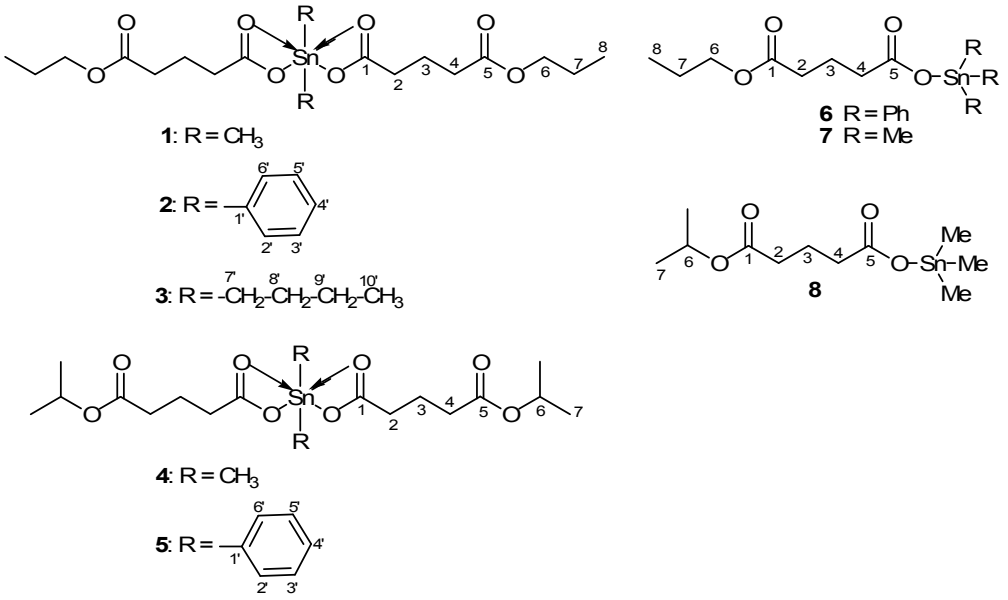


Figure 1. Proposed structures of di- and triorganotin(IV) complexes (**1-8**)

The frequency diffrences viz., $\nu\Delta$ (CO_2), in the diorganotin derivatives, **1-5** is smaller than the corresponding $\nu\Delta$ (CO_2) values of the triorganotin derivatives and this may attributed to steric crowding around the tin atom. Thus, for diorganotin compounds the $\nu\Delta$ (CO_2) values were found to be less than 200 cm-1 which indicates that the carboxylate groups are chelated and bonded to the metal in a bidentate manner [2,6].

Table 1. Infrared frequencies (cm^{-1}) of organotin derivatives.

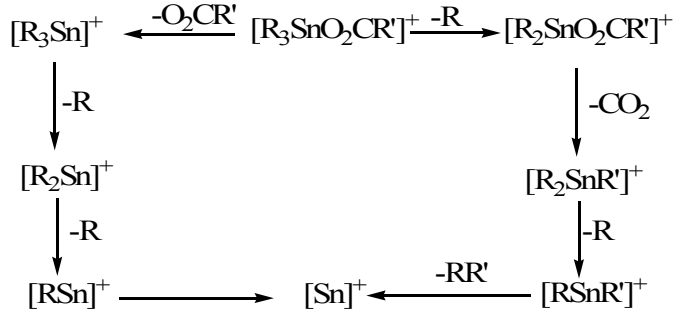
Compd.	νasym (CO_2)	νsym (CO_2)	Δν (CO_2)	ν(Sn-O)	ν(Sn-C)
1	1590 m	1415 m	175	450 m	535 s
2	1585 s	1405 m	180	463 m	520 m
3	1583 s	1415 s	168	453 s	540 w
4	1570 m	1405 s	165	455 s	533 m
5	1580 s	1407 m	173	450 m	532 s
6	1615 s	1345 s	270	455 m	519 s
7	1600 s	1350 m	250	465 m	537 w
8	1600 s	1350 m	250		

s: strong; m: medium; w: weak.

2.3. Mass Spectrometry

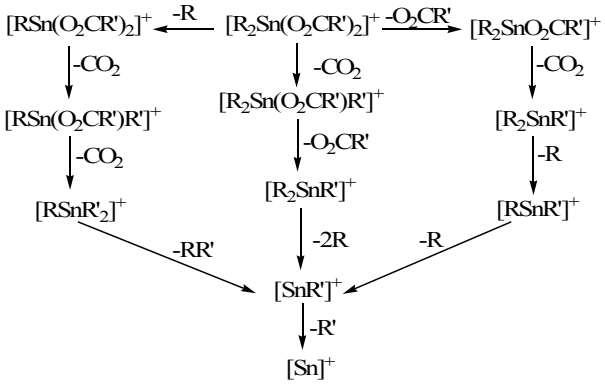
The molecular ion peak in almost all of derivatives was not observed. In the triorganotin (IV) derivatives **6-8** the major fragmentation observed is due to loss of the ligand moiety from the tin derivatives (Scheme 2; see Experimental) [2,3]. Successive fragmentation is observed by the loss of

"R" groups (Me, Bu, Ph) until the Sn^+ ion is obtained. In the alternative route "R" groups are eliminated first and in the next step a molecule of CO_2 is evolved from the ligand moiety attached to the tin atom. In the successive steps the remaining substituents are lost from the tin atom.



Scheme 2. General Mass fragmentation pattern for triorganotin(IV) carboxylates.

In the diorganotin (IV) derivatives fragmentation takes place according to the fragmentation pattern suggested in Scheme 3 (see Experimental) [2,3]. The main fragmentation observed is due to the loss of a ligand molecule followed by the loss of CO_2 from the second ligand. The successive loss of "R" groups and the loss of the remaining part of the ligand proceed until Sn^+ is obtained. An alternative route suggests that a loss of an "R" group occurs first followed by the successive loss of two molecules of CO_2 and then the remaining "R" groups. The third route proposed is the loss of a CO_2 molecule followed by the elimination of one ligand and then loss of the "R" groups.



Scheme 3. General Mass fragmentation pattern for diorganotin(IV) carboxylates

2.4.[1]H-NMR Spectroscopy

Chemical shifts for the various protons in the compounds are given in Experimental section. The conclusions drawn from the ^{1}H-NMR spectral studies lend further support to the mode of bonding discussed above. For instance, absence of signals between 10.00 and 13.00 ppm due to COOH protons confirms the deprotonation of the carboxylic acid oxygen atom of the ligand upon complexation. The protons of different organic groups viz., butyl, phenyl and methyl attached to the tin nucleus in these compounds appear at the appropriate positions in accordance to the previously reported values [2,7,8]. The number of protons of the various groups, calculated from the integration curves and those calculated for the expected molecular formulae are in complete agreement with each other.

2.5.[13]C-NMR Spectroscopy

The ^{13}C-NMR chemical shifts of various carbon atoms are given in Experimental section. The signals of the carbonyl carbon of the organotin complexes are observed at lower δ values upon complexation, compared to those of the uncomplexed ligands. The ^{13}C chemical shifts of methyl, butyl

and phenyl groups attached to tin are observed at similar positions compared with other, analogous compounds [2,7,8].

2.6.^{119}Sn NMR spectra

^{119}Sn-NMR chemical shifts of organotin(IV) compounds cover a range of roughly 600 ppm. The experimental data shows a significant shift of the resonance peaks to lower frequencies in proceeding from the starting organotin(IV) reagents to the 1:1 and 1:2 adducts, as reported for the analogous organotin(IV) derivatives [2,9,10]. These results are in agreement with the hypothesis of an increase in the coordination number of the tin atom in the complexes, and hence, of tin nuclear shielding [2,9,10].

3. Biological studies

Antibacterial activity: Antibacterial activity was evaluated using two Gram positive (*Bacillus subtilis, Staphlococcus aureus*) and four Gram-negative (*Escherichia coli, Schigella flexenari, Pseudomonas aeruginosa, Salmonella typh*) bacteria and the results are summarized in Table 2. In order to have a basis of comparison of our results, Imipinem was used as the standard drug. From the results in Table 2 it may be concluded that that diorganotin(IV) complexes are slightly less reactive than triorganotin(IV) complexes.

Table 2. Anti bacterial bioassay results [a,b,c,d] for R_2SnL and R_3SnL (Inhibition zone in mm)

Micro-organism	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	Std. drug
					Gram-positive				
Bacillus subtillis	10	14	13	12	16	24	20	19	43
Staphylococcus aureus	18	na	na	18	18	25	21	23	30
					Gram-negative				
Echerichia coli	18	na	na	18	18	25	21	23	30
Schigella flexenari	14	13	19	na	11	22	na	22	33
Psedomonas aeruginosa	14	15	20	14	na	20	18	21	25
Salmonella typh	18	20	14	19	18	22	21	20	41

[a]Concentration used: 1.00 mg/1.00 mL of DMSO; [b]size of well: 6 mm (diameter); [c]Standard drug: Imipinem; [d]na: no activity

4. Experimental

4.1. General experimental procedures

For general methods and instrumentation see ref. [2].

4.2. Synthesis of Ligands

A ten fold excess of the dry alcohols viz., isopropanol and *n*-propanol were separately added to glutaric anhydride and the subsequent mixtures were heated under reflux for 3 hours with constant stirring. Each reaction mixture was then evaporated under reduced pressure to remove the excess of the respective alcohol and the product half esters were dried in a desicator under reduced pressure.

4.3. Synthesis of Complexes:

4.3.1. The general method for the synthesis of diorganotin(IV) derivatives

Five new diorganotin(IV) compounds **1-5** were prepared as follows: For compounds **1**, **2, 4** and **5;** to a chloroform solution **(** 50 ml **)** of the ligands (0.01 mol) were added the diorganotin chloride (0.005 mol) and Et_3N (0.01 mol). The resultant mixture was heated under reflux with stirring for 3 hours and then allowed to stand at 25° C for 10 hours. The precipitated Et_3NH_4Cl salt that formed was filtered and the filtrate was evaporated under vacuum to yield a solid residue which was recrystalized from dichloromethane/hexane (1:1). For the synthesis of the one diorganotin(IV) compound **3,** the procedure was modified and involved a reflux period of 6 hours and the use of toluene as solvent and using a "Dean and Stark" apparatus.

4.3.2. The general method for the synthesis of triorganotin(IV) derivatives

Three new triorganotin(IV) compounds i.e. triphenyltin(IV) *n*-propyl glutrate (**6**), trimethyltin(IV) *n*-propyl glutrate (**7**), and trimethyltin(IV) monoisopropyl glutrate (**8**) were prepared as follows: To a solution of the carboxylate ligands (HL) (0.01 mol) in chloroform (50 ml) was added dry Et_3N (0.1 mol) followed by the addition of the triorganotin chloride (0.01 mol) in solid form at 25° C and the resulting mixture was heated under refux for 3 h. After cooling the reaction mixture to 25° C the precipitated Et_3NHCl was filtered off and the solvent was removed from the filtrate by a rotary evaporation. The solid residue was triturated with ethanol to afford the pure products.

Dimethyltin(IV) bis mono *n*-propyl glutarate (1): Solid; Yield: 84 %; mp 71 °C ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.06 (t, J = 7.0 Hz, 2H, H-6), 2.45 (m, 2H, H-2), 1.78 (m, 2H, H-7), 1.76 (m, 2H, H-3), 2.45 (m, 2H, H-4), 0.93 (t, J = 6.1 Hz, 3H, H-8), 0.91 [(s, 3H, Sn-Me) ^{2}J[(78.2]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 177.9 (C-1), 165.7 (C-5), 70.1 (C-6), 38.3 (C-2), 34.1 (C-4), 28.5 (C-3), 25.2 (C-7), 13.9 (C-8), 9.51 (C-9)Sn^{119} NMR ($CDCl_3$): –315MS (*m/z*, rel. int.): 481 (14%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]_2SnCH_3^+$), 466 (11%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]_2Sn^+$), 323 (12%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]Sn(CH_3)_2^+$), 293 (59%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]Sn^+$), 265 (5%, $[(CH_3(CH_2)_2CO_2(CH_2)_3]SnCH_3H^+$), 249 (26%, $[(CH_3(CH_2)_2CO_2(CH_2)_3]Sn^+$), 173 (78%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]^+$), 129 (29%, $[(CH_3(CH_2)_2CO_2(CH_2)_3]^+$); Anal. Calcd. for $C_{18}H_{32}O_8Sn$ (494): C, 43.66; H, 6.51. Found: C, 43.55; H, 6.40.

Diphenyltin(IV) bis mono *n*-propyl glutarate (2): Solid; Yield: 83 %; mp 74 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.10-7.73 (m, 5H, Ph), 4.03 (t, J = 7.0 Hz, 2H, H-6), 2.43 (m, 2H, H-2), 2.42 (m, 2H, H-4), 1.77 (m, 2H, H-7), 1.75 (m, 2H, H-3), 0.92 (t, J = 6.1 Hz, 3H, H-8); ^{13}C NMR (125 MHz, $CDCl_3$): δ 176.3 (C-1), 165.4 (C-5), 138.2 (C-1′), 129.9 (C-2′,C-6′), 129.8 (C-4′),129.7 (C-3′,C-5′), 70.3 (C-6), 38.1 (C-2), 34.3 (C-4), 28.3 (C-3), 25.5 (C-7), 13.1 (C-8). Sn^{119} NMR ($CDCl_3$): –305.5
MS (*m/z*, rel. int.): 543 (15%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]_2SnC_6H_5^+$), 466 (12%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]_2Sn^+$), 447 (14%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]Sn(C_6H_5)_2^+$), 327 (12%, $[(CH_3(CH_2)_2CO_2(CH_2)_3]SnC_6H_5H^+$), 293 (61%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]Sn^+$), 249 (28, $[(CH_3(CH_2)_2CO_2(CH_2)_3]Sn^+$), 173 (65%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]^+$), 129 (31%, $[(CH_3(CH_2)_2CO_2(CH_2)_3]^+$); Anal. Calcd. for $C_{28}H_{36}O_8Sn$ (620): C, 54.30; H, 5.86. Found: C, 54.35; H, 5.80.

Dibuyltin(IV) bis mono *n*-propyl glutarate (3): Solid; Yield: 83 %; mp 75 °C.^{1}H NMR (500 MHz, $CDCl_3$): δ 4.05 (t, J = 7.0 Hz, 2H, H-6), 2.47 (m, 2H, H-2), 2.47 (m, 2H, H-4), 1.77 (m, 2H, H-3), 1.75 (m, 2H, H-7), 1.72 (m, 2H, H-9′), 1.25-1.40 (m, 4H, H-7′, H-8′), 0.93 (t, J = 6.1 Hz, 3H, H-8), 0.85 (t, J = 6.1 Hz, 3H, H-10′); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.4 (C-1), 165.9 (C-5), 70.2 (C-6), 38.7 (C-2), 34.2 (C-4), 30.7 (C-8′), 28.1 (C-3), 26.6 (C-7′), 25.3 (C-7), 24.9 (C-9′), 13.5 (C-10′), 13.4 (C-8); Sn^{119} NMR ($CDCl_3$): –270 MS (*m/z*, rel. int.): 523 (17%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]_2SnC_4H_9^+$), 466

(12%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]_2Sn^+$), 407 (19%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]Sn(C_4H_9)_2^+$), 307 (9%, $[(CH_3)_2CHCO_2CH{=}CH]SnC_4H_9H^+$), 293 (61%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]Sn^+$), 249 (28, $[(CH_3(CH_2)_2CO_2(CH_2)_3]Sn^+$), 173 (65%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]^+$), 129 (31%, $[(CH_3(CH_2)_2CO_2(CH_2)_3]^+$); Anal. Calcd. for $C_{24}H_{44}O_8Sn$ (580): C, 48.18; H, 6.57. Found: C, 48.09; H, 6.66.

Dimethyltin(IV) bis *iso*propyl glutarate (4): Solid; Yield: 85 %; mp 72 °C. ^{1}H NMR (500 MHz, $CDCl_3$): δ 3.64 (m, 2H, H-6), 2.45 (m, 2H, H-4), 2.44 (m, 2H, H-2), 1.75 (m, 2H, H-3), 1.15 (d, J = 7.0 Hz, 6H, H-7), 0.93 [(s, 3H) ^2J[77.4]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 175.7 (C-1), 165.9 (C-5), 70.7 (C-6), 38.9 (C-2), 34.7 (C-4), 29.1 (C-3), 21.3 (C-7), 9.4 (Sn-Me). Sn^{119} NMR ($CDCl_3$): –325
MS (*m/z*, rel. int.): 481 (14%, $[(CH_3)_2CHCO_2(CH_2)_3CO_2]_2SnCH_3^+$), 466 (11%, $[(CH_3)_2CHCO_2(CH_2)_3CO_2]_2Sn^+$), 323 (12%, $[(CH_3)_2CHCO_2(CH_2)_3CO_2]Sn(CH_3)_2^+$), 293 (59%, $[(CH_3)_2CHCO_2(CH_2)_3CO_2]Sn^+$), 265 (5%, $[(CH_3)_2CHCO_2(CH_2)_3]SnCH_3H^+$), 249 (26%, $[(CH_3)_2CHCO_2(CH_2)_3]Sn^+$), 173 (78%, $[(CH_3)_2CHCO_2(CH_2)_3CO_2]^+$), 129 (29%, $[(CH_3)_2CHCO_2(CH_2)_3]^+$); Anal. Calcd. for $C_{18}H_{32}O_8Sn$ (494): C, 43.66; H, 6.51. Found: C, 43.55; H, 6.40.

Diphenyltin(IV) bis monoispropyl gulatrate (5): Solid; Yield: 81 %; mp 85 °C.^{1}H NMR (500 MHz, $CDCl_3$): δ 3.53 (m, 1H, H-6), 2.45 (m, 4H, H-2, H-4), 1.76 (m, 2H, H-3), 1.13 (d, J = 7.0 Hz, 6H, H-7), 7.10-7.73 (m, 5H, Ph); ^{13}C NMR (125 MHz, $CDCl_3$): δ 176.9 (C-1), 165.9 (C-5), 138.0 (C-1′), 129.7 (C-2′,C-6′), 129.1 (C-4′),128.7 (C-3′,C-5′), 70.5 (C-6), 38.7 (C-2), 34.4 (C-4), 28.7 (C-3), 21.7 (C-7).Sn^{119} NMR ($CDCl_3$): –300 MS (*m/z*, rel. int.): 543 (13%, $[(CH_3)_2CHCO_2(CH_2)_3CO_2]_2SnC_6H_5^+$), 466 (15%, $[(CH_3)_2CHCO_2(CH_2)_3CO_2]_2Sn^+$), 447 (13%, $[(CH_3)_2CHCO_2(CH_2)_3CO_2]Sn(C_6H_5)_2^+$), 327 (17%, $[(CH_3)_2CHCO_2(CH_2)_3]SnC_6H_5H^+$), 293 (60%, $[(CH_3)_2CHCO_2(CH_2)_3CO_2]Sn^+$), 249 (28, $[(CH_3)_2CHCO_2(CH_2)_3]Sn^+$), 173 (63%, $[(CH_3)_2CHCO_2(CH_2)_3CO_2]^+$), 129 (33%, $[(CH_3)_2CHCO_2(CH_2)_3]^+$); Anal. Calcd. for $C_{28}H_{36}O_8Sn$ (620): C, 54.30; H, 5.86. Found: C, 54.35; H, 5.80.

Triphenyltin(IV) mono *n*-propyl glutrate (6):Solid; Yield: 84 %; mp 88-89 °C.^{1}H NMR (500 MHz, $CDCl_3$): δ 4.06 (t, J = 7.0 Hz, 2H, H-6), 2.41 (m, 4H, H-2, H-4), 1.77 (m, 2H, H-7), 1.67 (m, 2H, H-3), 0.91 (t, J = 6.1 Hz, 3H, H-8), 7.13-7.89 (m, 15H, 3Ph); ^{13}C NMR (125 MHz, $CDCl_3$): δ 178.1 (C-1), 168.7 (C-5), 138.7 (C-1′), 129.9 (C-2′,C-6′), 129.3 (C-4′),128.3 (C-3′,C-5′), 70.4 (C-6), 35.9 (C-2), 35.1 (C-4), 30.1 (C-3), 25.7 (C-7), 13.5 (C-7).
Sn^{119} NMR ($CDCl_3$): –165
MS (*m/z*, rel. int.): 447 (6%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]Sn(C_6H_5)_2^+$), 403 (28%, $[(CH_3(CH_2)_2CO_2(CH_2)_3]Sn(C_6H_5)_2^+$), 326 (17%, $CH_3(CH_2)_2CO_2(CH_2)_3]SnC_6H_5^+$), 293 (29%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]Sn^+$), 249 (28, $[(CH_3(CH_2)_2CO_2(CH_2)_3]Sn^+$), 173 (65%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]^+$), 129 (31%, $[(CH_3(CH_2)_2CO_2(CH_2)_3]^+$); Anal. Calcd. for $C_{26}H_{28}O_4Sn$ (524): C, 59.69; H, 5.39. Found: C, 59.59; H, 5.27.

Trimethyltin(IV) mono *n*-propyl glutrate (7): Solid; Yield: 78 %; mp 88 °C.^{1}H NMR (500 MHz, $CDCl_3$): δ 4.03 (t, J = 7.0 Hz, 2H, H-6), 2.49 (m, 4H, H-2, H-4), 1.79 (m, 2H, H-3), 1.75 (m, 2H, H-7), 0.92 (t, J = 6.1 Hz, 3H, H-8), 0.92 [(s, 3H, Sn-Me) ^2J[77.7]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 178.9 (C-1), 168.9 (C-5), 70.0 (C-6), 36.8 (C-2), 36.5 (C-4), 29.8 (C-3), 25.8 (C-7), 13.4 (C-8); 9.7 (Sn-Me).Sn^{119} NMR ($CDCl_3$): –150 MS (*m/z*, rel. int.): 323 (18%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]Sn(CH_3)_2^+$), 279 (27%, $[(CH_3(CH_2)_2CO_2(CH_2)_3]Sn(C_6H_5)_2^+$), 264 (13%, $[(CH_3(CH_2)_2CO_2(CH_2)_3]Sn(CH_3)^+$), 293 (61%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]Sn^+$), 249 (26%, $[(CH_3(CH_2)_2CO_2(CH_2)_3]Sn^+$), 173 (31%, $[(CH_3(CH_2)_2CO_2(CH_2)_3CO_2]^+$), 129 (49%, $[(CH_3(CH_2)_2CO_2(CH_2)_3]^+$); Anal. Calcd. for $C_{11}H_{22}O_4Sn$ (338): C, 39.20; H, 6.58. Found: C, 39.29; H, 6.45.

Trimethyltin(IV) mono *iso*propyl glutrate (8): Solid; Yield: 80 %; mp 87 °C.^{1}H NMR (500 MHz, $CDCl_3$): δ 4.07 (t, J = 7.0 Hz, 2H, H-6), 2.45 (m, 2H, H-4), 2.43 (m, 2H, H-2), 1.79 (m, 2H, H-3), 1.75 (m, 2H, H-7), 0.92 (t, J = 6.1 Hz, 3H, H-8), 0.92 [(s, 3H, Sn-Me) ^2J[77.9]; ^{13}C NMR (125 MHz, $CDCl_3$): δ 178.9 (C-1), 168.8 (C-5), 71.0 (C-6), 36.8 (C-2), 36.5 (C-4), 29.9 (C-3), 20.8 (C-7), 9.55 (Sn-Me).Sn^{119} NMR ($CDCl_3$): –160MS (*m/z*, rel. int.): 323 (18%,

$[(CH_3)_2CHCO_2(CH_2)_3CO_2]Sn(CH_3)_2^+$), 279 (27%, $[(CH_3)_2CHCO_2(CH_2)_3]Sn(CH_3)_2^+$), 264 (13%, $[(CH_3)_2CHCO_2(CH_2)_3]Sn(CH_3)^+$), 293 (61%, $[(CH_3)_2CHCO_2(CH_2)_3CO_2]Sn^+$), 249 (26%, $[(CH_3)_2CHCO_2(CH_2)_3]Sn^+$), 173 (31%, $[(CH_3)_2CHCO_2(CH_2)_3CO_2]^+$), 129 (49%, $[(CH_3)_2CHCO_2(CH_2)_3]^+$); Anal. Calcd. for $C_{11}H_{22}O_4Sn$ (338): C, 39.20; H, 6.58. Found: C, 39.29; H, 6.45.

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