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Biologically potent organotin(IV) complexes of 2-maleimidoacetic acid

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Abstract

The in vitro LD_{50}, anti-bacterial, anti-fungal and anti-yeast bio-tests are carried out, which proved them to be powerful biocides. The in vitro anti-tumour and analgesic activities also displayed excellent potential of the title compounds. Series of organotin(IV) complexes are synthesized and characterized. Based on spectroscopic analysis and literature evidences, the compound 1 is tetrahedral and 2 distorted octahedral or trigonal bipyramidal in nature wherein the compounds 3 and 4 are tetrahedral in solid and polymeric trigonal bipyramidal geometry in solution. Beside to ^1H, ^{13}C and ^{119}Sn NMR, the FT-IR is successfully applied to verify the bonding mode of *endo* and *exo* status of tin(IV) of compound 2.
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1. Introduction

Organotin(IV) complexes of amino acids and their organic derivatives containing the carboxylic O–Sn(IV) bond, display significant anti-tumour activity and promising potential in many other fields like, wood preservation, polymer chemistry, pesticidal, bactericidal and anti-fouling agents, etc. [1–4]. The structural chemistry of organotin(IV) complexes of carboxylic moieties as amino acids and *N*-protected amino acids with a coordination number higher than four is being extensively studied because of their biological activity, enhanced reactivity and stereochemical non-rigidity [5–9].

Several reports have been cited in the literature by other research groups pertaining the bioactivity as anti-tumour agents and structural chemistry of di- as well as triorganotin(IV) compounds [5–10]. This is an extension of our previous work on bioactivity and structural chemistry of organotin(IV) complexes of *N*-phthaloy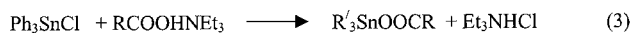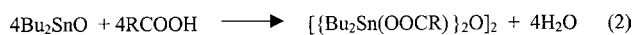la-mino acids [9]. In the present work 2-maleimidoacetic acid has been selected as a ligand on account of its importance as a bioactive molecule as reported in the literature [11]. The literature reveals that almost no work has been carried out over 2-maleimidoacetic acid in spite of the fact it show a number of bioactivities. Keeping in view these facts, we want to extend our work by introducing maleimido group and to explore the bioactivities of the compounds. We here report the biological screening tests, synthesis and spectroscopic characterization of the novel title complexes. The complexation of *N*-maleimidoacetic acid with tin(IV) metal yield products of exceedingly improved character as powerful biocides.
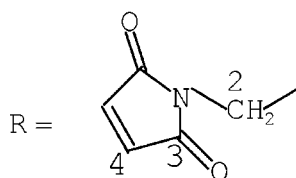
2. Results and discussion

2.1. Syntheses

The 2-maleimidoacetic acid was synthesized as reported [11]. The synthesis of di-*n*-butyltin(IV)di-2-maleimidoacetate, compound 1, bis[di-*n*-butyl(2-maleimidoacetato)-tin(IV)] oxide, compound 2, tri-phenyl-tin(IV)-2-maleimidoacetate, compound 3 and tri-

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$$Bu_2SnO + 2RCOOH \longrightarrow Bu_2Sn(OOCR)_2 + 2H_2O \quad (1)$$

$$4Bu_2SnO + 4RCOOH \longrightarrow [\{Bu_2Sn(OOCR)\}_2O]_2 + 4H_2O \quad (2)$$

$$Ph_3SnCl + RCOOHNEt_3 \longrightarrow R'_3SnOOCR + Et_3NHCl \quad (3)$$

$$Cyc_3SnCl + RCOOHNEt_3 \longrightarrow R'_3SnOOCR + Et_3NHCl \quad (4)$$

(Where: Ph = Phenyl & Cyc = Cyclohexyl)

Scheme 1.

cyclohexyltin(IV)-2-maleimidoacetate, compound 4, is described in Scheme 1.

2.2. Spectral studies of mono and dimer complexes

The OH absorption of ligand disappeared in the complexes. Both asymmetric and symmetric stretching of the maleimido (C_2O_2N), the carbonyl (CO), for Sn–C and Sn–O groups were exhibited as reported in literature [12,13]. The asymmetric and symmetric stretching of the CO group in monomer exhibit trend like a: $v_{asym (compound)} > v_{asym (ligand)}$, $v_{sym (compound)} < v_{sym (ligand)}$, $\Delta v_{(compound)} > \Delta v_{(ligand)}$. The monomer tin ester shows unidentate or weak bidentate bonding with Sn(IV) atom. The order of asymmetric and symmetric of CO group of compound 2 with respect to ligand is as: $v_{asym (compound)} < v_{asym (ligand)}$, $v_{sym (compound)} < v_{sym (ligand)}$, $\Delta v_{(compound)} > \Delta v_{(ligand)}$. In this case, two types of CO absorption bands were observed at 1685–1372 cm^{-1} for bonding behavior and 1714–1695 cm^{-1} for non-bonding behavior, which indicate two tin sites of compound 2. The coordination of the *endo* and *exo* status of tin in dimeric compound 2 is successfully described on the basis of IR spectral data. Two intensive absorption bands for carbonyl linked to Sn(IV) are observed which confirmed two non-equivalent tin spheres. No literary evidence was found for such exclusive structural description about the similar compounds on the basis of IR study. The butyl protons in mono and dimer were resolved on proper positions as reported [14–16]. The methyl protons of monomer exhibits a single triplet, which indicate one tin site, and two triplets of methyl protons in compound 2 are due to non-equivalent status of methyl protons bonded to *endo* and *exo* tin(IV) atoms. The ^{13}C NMR signals are properly resolved, show one signal for each methylene carbon in butyl group monomer and a pair of signals in dimer tin ester around *exo* and *endo* cyclic Sn(IV), which are easily identified from their $^nJ(^{119}$Sn–^{13}C) coupling constants for both the mono and dimer compounds with well known ranking

$[^1J] \gg [^3J] > [^2J]$ [17–20]. The ^{119}Sn NMR data of compound 1 exhibits single resonance at −151.6 ppm, which suggests tetrahedral status in solution [6]. The compound 2 is characterized by two signals of equal intensities (−214.5/−216.4) for *endo*- and *exo*-cyclic tin atoms, which indicates the distorted octahedral geometry [8]. The Mössbauer parameters are found in the range of 3.41 mm s^{-1} for compound 1 which is greater than 2.1 for a trans octahedral geometry around tin atom of monomer in solid (Fig. 1) [14,15,17–20]. The large quadrupole splitting value (QS = 3.65 mm s^{-1}) for dimer, compound 2, recommends penta-coordinate environment in solid state, which suggests the tetrabutyl bis(2-maleimidoacetato) distannoxane dimer type (Fig. 2). The two different tin atoms could not be discriminated by Mössbauer spectroscopy since the method is less sensitive to small variations to the tin environment than $^{119/117}$Sn NMR spectroscopy [8]. The ^{1}H, ^{13}C, ^{119}Sn NMR, QS and FT-IR data of compounds 1 and 2 are in agreement with the structure proposed in Figs. 1 and 2. The percentage CHN analysis verifies the mono and dimeric composition of compounds 1 and 2.

2.3. Spectral studies of triorganotin(IV) complexes

The order of CO stretching of compounds 3 and 4 with respect to ligand is like as: $v_{asym (compound)} < v_{asym (ligand)}$,

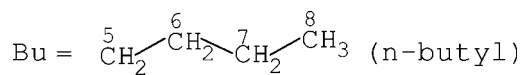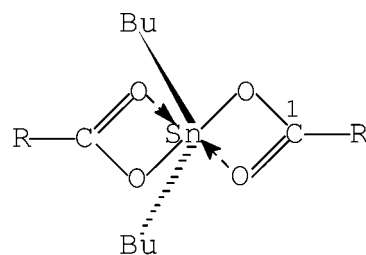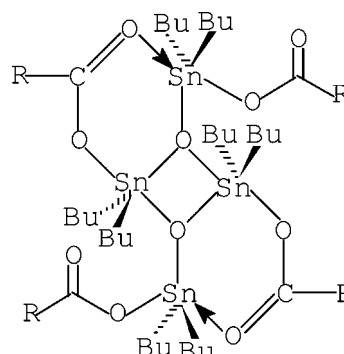Fig. 1.

Fig. 2.

$v_{\text{sym (compound)}} < v_{\text{sym (ligand)}}$, $\Delta v_{\text{(compound)}} > \Delta v_{\text{(ligand)}}$. The shifting of CO band for compound 3 at 1680_{asym} and 1356_{sym} and for compound 4 at 1695_{asym} and 1378_{sym} indicate the penta-coordinate structure in solid [16] (Fig. 4). Such kind of coordination could not be seen during the ^{1}H, ^{13}C, and ^{119}Sn NMR spectroscopic analyses in inert solvent but exhibit four-coordinate monomeric species consistently as suggested for comparable systems [21–23]. The expected NMR resonance is assigned by their multiplicity, intensity and their coupling constants pattern. The exhibited $^1J(^{119}$Sn–^{13}C$)$ coupling constants in solution are characteristic of tetrahedral compounds, being of the order of 662 Hz for triphenyltin(IV) and 346 Hz for tricyclohexyltin(IV) esters [9]. The ^{119}Sn NMR signals are in the range of -116.3 ppm for Compound 3 and 8.6 ppm for the Compound 4 recommends also four-coordinate environment around tin(IV) atom. Based on the spectroscopic and literature evidences, the compounds 3 and 4 lead to polymeric trigonal bipyramidal geometry in solid and tetrahedral geometry in inert solvent [13,22]. The ^{119m}Sn Mössbauer of Compounds 3 and 4 exhibit quadrupole splitting values of 3.42 and 3.63 mm s^{-1} respectively. Based on reported literature the compounds with QS values 3.59–3.70 mm s^{-1} have five coordinate chain structure, trigonal bipyramidal geometry with a bridging carboxyl group in solid [9,21] and displayed a tetrahedral geometry in an inert solvent (Figs. 3 and 4) [3,6,21]. This leads to slight contradiction with Sharma, who found ^{119}Sn resonance at -116 ppm and a QS value of 2.44 mm
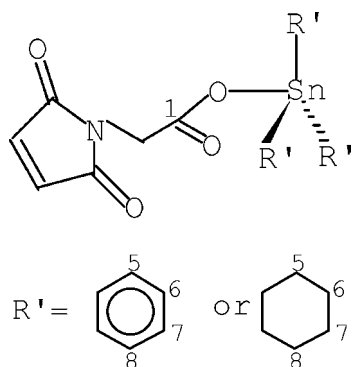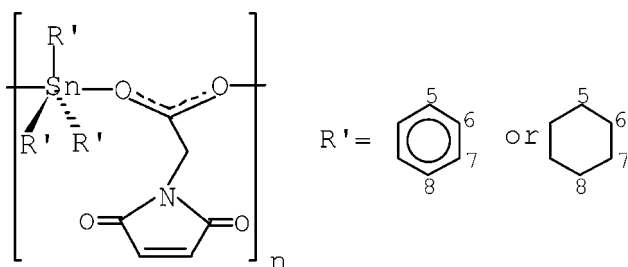
s^{-1} for $[(C_6H_5)_3Sn(O_2CCH{=}CH{-}C_6H_4OCH_3{-}p)]$. The $\delta = QS/IS$ also supports the same geometry [9,21]. Such type of structural similarity between triphenyltin(IV) and tricyclohexyltin(IV) derivatives have not been observed. The pinpoint accuracy for bond angles and bond lengths for the novel title complexes can only be achieved and reported on the bases of XRD data. The X-ray studies of compounds 1–4 could not be carried out due to lack of facility.

3. Biological activities

All the compounds exhibited highest toxicity but 4, is more potent against Brine shrimp larvae (Table 1). In vitro significant phytotoxic anti-tumour activity (Table 2) for compounds 1–4 indicating their biological importance while reported organotin(IV) derivatives of *N*-phthaloylamino acids did not show such properties [10]. The analgesic behavior is measured against acetic acid treated mice (Table 3). During the investigation an outstanding analgesic response was observed and found more effective than ligand 5, which may be due to the presence of tin metal. It may be considered a step towards the biological and clinical role of these compounds. While such results were not followed in our previous work [10]. All the compounds (1–4) exhibited strong bactericidal and fungicidal properties, see Tables 4 and 5. Among such compounds, the carboxylate derivatives are used as anti-cancer, anti-tumour agents, fungicides or bactericides in vitro as well as in vivo [23–25]. The biocidal activity of triorganotin(IV) motifs is enhanced on account of their geometric behavior in solution. The tetrahedral structure in solution is more active than other forms. It is also reported in the literature [7,21,26,27] that the four-coordinated motifs has stronger tendency to increase the coordination numbers through O, S, or N donor groups while the five coordinated species do not undergo further coordination, which play no long term role in the vivo chemistry of organotin(IV) esters. Triorganotin(IV) class is significantly active than other classes due to a greater partition

Fig. 3.

Fig. 4.

Table 1
Brine shrimp bioassay

Compounds	Deaths at doses (%)			LD$_{50}$ (µg/ml)	Results
	0.1 µg/ml	0.05 µg/ml	0.025 µg/ml		
1	96	73	83	0.0483	++++
2	76	46	76	0.0349	++++
3	100	73	53	0.0204	++++
4	100	90	90	0.008	++++
5	60	20	10	616.77	++

Key: ++++, highest lethality; ++, positive lethality.

Table 2
In vitro phytotoxic (anti-tumour) bioassay[a]

Compounds	Dose (µg/ml)	No. of fronds		Growth regulation (%)	F1 50[b] (µg/ml)
		Experimental	Control		
1	500	00	11	100	0.0000
	50	0	9	100	0.125
	5	8	9	11	
2	500	00	11	100	0.0000
	50	00	9	100	0.125
	5	7	10	30	
3	500	00	11	100	0.0000
	50	00	11	100	0.095
	5	00	11	100	
4	500	00	11	100	0.0000
	50	00	11	100	0.095
	5	00	11	100	
5	500	3	11	72	0.0000
	50	5	11	55	0.111
	5	7	11	36	

[a] Lemma acqunioctialis welv.
[b] Concentration used to inhibit and 20 promote 50% of frond proliferation. Reference inhibitor: praqual 100%.

Table 3
Analgesic activity on albino mice

Dosagemg (Kg)	No. of writhes[a]	Percentage of inhibition by compounds					Aspirin[b]
		1	2	3	4	5	
Control	62						–
10		11 (55)	9 (56)	16 (52)	18 (51)	10 (56)	–
50		31 (43)	28 (45)	45(35)	55 (28)	45 (34)	–
100		77 (14)	65 (22)	63 (23)	82 (11)	55 (28)	–
150							65(40)

Note. Writhes remained are given in parentheses.
[a] Acetic acid.
[b] Standard drug.

Table 4
Bactericidal bioassay

Name of bacteria	Activity of compounds				
	1	2	3	4	5
Human pathogens					
Acillus cereus	+++	++++	+++	+++	+
Bacillus subtilis	++++	+++	+++	++++	++
Cornyebacterium diphtheriae	+	+++	+++	+++	+
Escherichia coli ETEC	++	++	++++	++++	++
Klebsiella pneumoniae	++	+++	++++	++++	++
Salmonella typhi	++	+	++	++++	+
Staphylococcus aureus	+++	+++	++++	+++	+
Shigella boydii	++	+++	++++	+++	+++

Key: ++++, highest; +++, high; ++, optimum; +, no activity. Incubation period: 8 h, 37 °C, colony forming unit, 10^4–10^6; size of well, 5 mm radius. Reference drug: Amoxicillin (H_2O), Ampicillin(H_2O), Cephlaxin Na.

coefficient value [7]. The results of the anti-yeast bioassay were not very encouraging (Table 6).

4. Conclusions

Notable geometrical trends on the bases of IR data and coupling constant patterns are observed for compounds 1–4 in relationship to our previous work and reported by other groups [10]. The monomer showed only one sharp ^{119}Sn NMR signal while the dimer exhibited two signals due to *exo* and *endo* Sn(IV) atoms. The IR shifting of CO group in monomer supports weak bidentate coordination to Sn(IV), which also gives clue about its octahedral geometry in solid, which is also supported by Mössbauer data. The ^{119}Sn NMR spectrum of dimer type showed two signals of equal intensity. The *exo* tin(IV) atom may be bonded in monodentate mode with carboxylic oxygen of ligand, on the other hand the *endo* cyclic tin(IV) atom in bidentate mode. This mode of coordination with *exo* cyclic tin atoms results in penta coordination, trigonal bipyramidal geometry [16] (Fig. 5). Based on these results, we report here the ladder type view of the dimer ester

Table 5
Fungicidal bioassay

Name of fungi	Activity of compounds				
	1	2	3	4	5
Aspergillus flame	+	+	++++	++++	++
Human pathogens[a]					
Trichophyton schoenlem	++	++	++++	+++	+
Pseudallescheria boydii	++	++	+++	++++	++
Aspergillus niger[b]	+	+	++++	++++	+
Animal pathogens[a]					
Microsporum canis	–	–	–	–	–
Trichophyton mantagrophytes	+++	+++	++++	++++	+++
Trichophyton rubrum	+++	++	++++	+++	+
Trichophyton	+++	+++	++++	++++	++
Plant pathogens[c]					
Fusanum oxysponumvarlycopersici	++++	++++	++++	++++	++
Fusanum solanivarcopersici	++++	++	+++	++++	+++
Macrophormina phaseolina	++++	++++	++++	++++	+
Rhizoctonia solani	++++	++++	++++	++++	++

Key: ++++, highest; +++, high; ++, optimum; +, no activity. Incubation time = 7 h, 27 °C.
[a] Miconazole, Ketoconazole.
[b] AmphotenicinB, Flycytosine.
[c] Benlate, Nabam.

Table 6
Anti-yeast bioassay

Name of yeast[a]	Activity					Streptonigrin
	1	2	3	4	5	
(m).RS 322Y (RAD52)	+	++	++	+++	+	++++
(w). LF 15 (RAD$_+$)	++	++	+++	+++	++	++++

Key: ++++, very high; +++, high; ++, optimum.
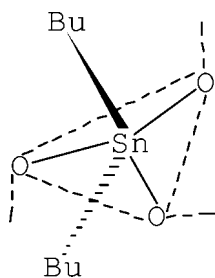[a] *Saccharomyces cerevisiae.*

Fig. 6.

Fig. 5.

(Fig. 6). Similarly, the tetrahedral geometry is assigned to triorganotin(IV) monomers in inert solvent and polymeric trigonal bipyramidal in solid state.

The toxic behavior of compounds 1–4 may be considered due to the non-involvement of the lone pair on oxygen atom of carbonyl group of ligand in the coordina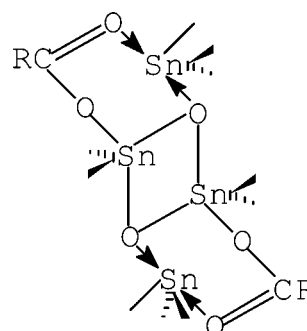tion process in solution, which may play a key role for escalation of toxicity. Since the lone pair may actually be bound through a weak/strong bidentate bonding in solid, which get free in solution form and provide most suitable site for coordination. Moreover, toxicity is also confined with an attached "R" group to tin(IV) atom. In the R_3SnL unit, the function of L (ligand) plays a key role in transporting the active organotin(IV) moiety to an action site, which is released on hydrolysis [7,28].

5. Experimental

5.1. Materials

Maleic anhydride, aminoacetic acid, di-*n*-butyltin(IV) oxide, triphenyltin(IV) chloride, tricyclohexyltin(IV) chloride and triethylamine are Merck Chemicals used without further purification. The ligands were prepared

according to the reported procedures [11]. The monomer compound 1 was prepared by dissolving 1.00 g (4.0 mmol) dibutyltin oxide to 8.0 mmol ligand in 150 cm^3 of toluene and 50 cm^3 ethanol. The mixture was refluxed for 6 h and the ternary azeotrope water/ethanol/toluene was distilled off with Dean-Stark funnel. Half of remaining solution was evaporated under vacuum. The oily compound obtained was crystallized from mixture (4:1) of chloroform and hexane. The synthesis of compound 2 occurs similarly but half the amount of the ligand is used, i.e., 4.0 mmol. The triorganotin(IV) complexes were synthesized by the refluxing the triethylammonium salt of ligand [11] with tri-phenyltin(IV) chloride or tri-cyclohexyltin(IV) chloride in toluene for 3 h. The precipitated triethylammonium hydrochloride formed in this reaction was filtered off. The solvent was removed under vacuum and the solid mass left was crystallized from dichloromethane.

5.2. Physical measurements

A Reichert Thermovar of F.G. Bode Co. Austria was used to measure the melting points. FT-IR spectra were obtained using KBr disc on a Perkin–Elmer FTIR1600 Spectrometer. Elemental analyses were carried out on a Yanaco MT 3 high-speed CHN analyzer with an antipyrene as a reference compound. The ^{1}H NMR spectra were recorded on a multinuclear FT-NMR 200 MHz of JEOL and ^{13}C spectra were taken at 50 MHz using a ^{13}C probe. The ^{119}Sn NMR spectra were obtained 93.28 MHz with ^{119}Sn probe. The TMS and $(CH_3)_4$Sn were used as internal standards. The ^{119m}Sn Mössbauer spectra were recorded on V.G. Micromass 7070 F instrument at a temperature of 200 °C.

LD$_{50}$ of the complexes was determined against a brine shrimp hatching method [29] while bactericidal and fungicidal activities were measured by agar-well diffusion and agar-tube dilution methods [30]. In vitro phytotoxic anti-tumour activity tests (potato disc assay) were carried out using fronds of Lemna acquinoctialis welv for the purpose [30,31]. Albino mice were under the experiments for an analgesic activity test and a number of writhing responses for each animal were recorded using aspirin as a standard drug. Acetic acid was used for writhing response. Intraperitoneal route was used for the dose administration [32,33]. The anti-yeast bioassay of ligand and the organotin(IV) compounds against mutant (*Saccharomyces cerevisiae*, Rs 322Y RAD52) and wild type strains (*Saccharomyces cerevisiae* LF15RAD$_+$) was determined. Streptonigrin was used as a standard drug.

5.3. Spectroscopic results

Mössbauer data: QS, quadrupole splitting; IS, isomer shift all in mm s^{-1}.

NMR data: all spectra were acquired from CDCl$_3$.

Abbreviations for coupling patterns: s, singlet; d, doublet; t, triplet; td, triplet of doublets; tq, triplet of quartets; b, broad resonance; m, complex pattern; nv, non-visible; coupling constants are given in hertz between parenthesis for $^nJ(^{119}$Sn–^{13}C).

Abbreviations for IR: asym, asymmetric; b, broad; s, small; sp, sharp; sym, symmetric; w, weak.

5.3.1. Compound 1 [C₂H₂(CO)₂NCH₂COO]₂SnBu₂

Recrystallized from chloroform, mp: >350 °C, yield: 83%, solubility: soluble in chloroform, methanol and insoluble in water, when four fold water was added, its alcoholic solution remained clear.

CHN analysis (%) antipyrene: C: 44.31 (44.39), H: 4.81 (4.84), N: 5.13 (5.17), the calculated values are in the parenthesis.

FT-IR analysis (cm^{-1}): C$_2$O$_2$N (maleimido): 1775$_{asym}$ sp, 1721$_{sym}$ ssp; CO(carbonyl): 1720$_{asym}$ sp, 1380$_{sym}$ sb; Δv : 340; Sn–C: 525 wsp; Sn–O: 509 wsp.

^{1}H NMR: H2: 4.23 s; H4: 7.43 s; H5: 1.64 t (7); H6: 1.52–1.61 m; H7: 1.33–1.50 m; H8: 0.91 t (7).

^{13}C NMR: C1: 177.5; C2: 41.2; C3: 168.2; C4: 129.0; C5: 28.1 [$^1J(^{119}$Sn–^{13}C) = 589]; C6: 31.4 [$^2J(^{119}$Sn–^{13}C) = 39]; C7: 26.8 [$^3J(^{119}$Sn–^{13}C) = 109]; C8: 13.2.

^{119}Sn NMR (CH$_3$)$_4$Sn: −151.6.

^{119m}Sn Mössbauer data (mm s^{-1}): QS: 3.41; IS: 1.35; G1: 1.18; G2: 1.17; δ = QS/IS: 2.53.

5.3.2. Compound 2 [{C₂H₂(CO)₂NCH₂COOSnBu₂}₂O]₂

Recrystallized from benzene/chloroform, mp: 158 °C, yield: 93%, solubility: soluble in CHCl$_3$ ethanol (on heating), insoluble in methanol and water.

CHN analysis (%) antipyrene: C: 42.53 (42.56), H: 5.06 (5.01), N: 0.36 (0.38).

FT-IR analysis (cm^{-1}): C$_2$O$_2$N (maleimido): 1774$_{asym}$ sp, 1720$_{sym}$ ssp; CO(carbonyl): 1685$_{asym}$ sp, 1372$_{sym}$ sb Δv : 313/1714$_{asym}$ ssp,1695$_{sym}$ sp; Sn–C: 530 wsp; Sn–O: 485 sp.

^{1}H NMR: H2: 4.21 s; H4: 7.38 s; H5: 1.50 t (8); H6: 1.44–1.48 m; H7: 1.20–1.25 m; H8: 0.94 t (7)/0.79 t (7).

^{13}C NMR: C1: 176.2; C2: 40.9; C3: 167.2; C4: 129.07; C5: 29.8/28.6 [$^1J(^{119}$Sn–^{13}C) = 684, nv]; C6: 26.2/26.8 [$^2J(^{119}$Sn–^{13}C) = 44, 43]; C7: 27.3/26.1 [$^3J(^{119}$Sn–^{13}C) = 131,129]; C8: 13.4/13.1.

^{119}Sn NMR (CH$_3$)$_4$Sn: −214.5, −216.4.

^{119m}Sn Mössbauer data (mm s^{-1}): QS: 3.65; IS: 1.32; G1: 0.84; G2: 0.82; δ = QS/IS: 2.77.

5.3.3. Compound 3 [(C₆H₅)₃SnOOCCH₂N(CO)₂C₂H₂]

Recrystallized from dichloromethane, mp: 139 °C, yield: 96%, solubility: soluble in chloroform, ethanol, slightly soluble in methanol and insoluble in water. When eight fold of water was added its ethanolic solution remained clear.

CHN analysis (%) antipyrene: C: 61.01 (61.05), H: 4.00 (4.05), N: 6.74 (6.77).

FT-IR analysis (cm^{-1}): C_2O_2N (maleimido): 1772_{asym} sp, 1715_{sym} ssp; CO (carbonyl): 1680_{asym} s, 1356_{sym} s; Δv : 324; Sn–C: 540 sp; Sn–O: 511 w.

^{1}H NMR: H2: 4.25 s; H4: 7.28 s; H6: 7.31– 7.34 m; H7/H8: 7.48–7.52 m.

^{13}C NMR: C1: 178.2; C2: 39.7; C3: 167.5; C4: 129.0; C5: 138.6 [$^1J(^{119}Sn–^{13}C) = 662$]; C6: 127.25 [$^2J(^{119}Sn–^{13}C) = 49$]; C7: 129.3 [$^3J(^{119}Sn–^{13}C) = 47$]; C8: 137.4 [$^4J(^{119}Sn–^{13}C) = 14$].

^{119}Sn NMR ($CH_3)_4$Sn: −116.3.

^{119m}Sn Mössbauer data (mm s^{-1}): QS: 3.42; IS: 1.6; G1: 0.84; G2: 0.81; $\delta = QS/IS$: 2.14.

5.3.4. Compound 4 [$(C_6H_{11})_3SnOOCCH_2N(CO)_2C_2H_2$]

Recrystallized from dichloromethane, mp: 117 °C, yield: 89%, solubility: soluble in chloroform and ethanol (on heating), slightly soluble in methanol but insoluble in water. When six fold water was added, its alcoholic solution remained clear.

CHN analysis (%): C: 58.72 (58.79), H: 7.50 (7.54), N: 2.82 (2.85).

FT-IR analysis (cm^{-1}): C_2O_2N (maleimido): 1772_{asym} sb, 1702_{sym} sb; CO(carbonyl): 695_{asym} ssp, 1378_{sym} ssp; Δv: 317; Sn–C: 557 sp; Sn–O: 518 w.

^{1}H NMR: H2: 4.22 s; H4: 7.1 s; H5: 1.47 t (7); H6– H8: 1.44–1.52 m.

^{13}C NMR: C1: 177.3; C2: 35.2; C3: 169.2; C4: 129.00; C5: [$^1J(^{119}Sn–^{13}C) = 346$]; C6: 33.4 [$^2J(^{119}Sn–^{13}C) = 16$]; C7: 29.6 [$^3J(^{119}Sn–^{13}C) \approx 15$]; C8: 28.7 [$^4J(^{119}Sn–^{13}C) = nv$.

^{119}Sn NMR ($CH_3)_4$Sn: 8.6.

^{119m}Sn Mössbauer data (mm s^{-1}): QS: 3.63; IS: 1.45; G1: 0.98; G2: 1; $\delta = QS/IS$: 1.88.

5.3.5. Compound 5 ligand: [$C_2H_2(CO)_2NCH_2COOH$]

Recrystallized from chloroform, mp: 114 °C, yield: 94%, solubility: soluble in ethanol and methanol, insoluble in chloroform and water.

CHN analysis (%) antipyrene: C: 46.39(46.42), H: 3.21 (3.24), N: 9.00(9.02).

FT-IR analysis (cm^{-1}): OH (acidic) 3435–3243 b, C_2O_2N (maleimido): 1781_{asym} mb; 1720_{sym} sb; CO (carbonyl): 1710_{asym} mb, 1692_{sym} ssp; Δv: 18.

Acknowledgements

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