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Organotin(IV) Complexes of 4,5-Dimethoxy-2-Nitrobenzoic Acid: Synthesis, Characterization, and Biological Activity[1]

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Abstract—A new series of organotin esters has been derived from the condensation of organotin oxides/halides with 4,5-dimethoxy-2-nitrobenzoic acid. Their spectroscopic investigations have been carried out both in solution and solid state. Experimental details for the preparation and the structural characterization (by FTIR, NMR, XRD, and EI mass spectral analysis) are provided. Based on spectroscopic results, the ligand appeared to coordinate to the Sn atom through the COO moiety. Single crystal analysis has shown a bridging behavior of ligand in tributyltin(lV) derivative. Bioassay results have shown that these compounds have good antibacterial, antifungal and cytotoxicity activity, with few exceptions.

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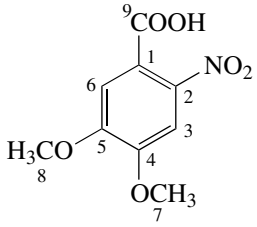
INTRODUCTION

Organotin carboxylates attract considerable attention due to their wide range of applications. Extensive use of organotin compounds in various fields of life results in quantum leap of organotin chemistry. Their stability and structure diversity make their coordination chemistry very interesting [1]. Organotin derivatives act as potentially active biological agents and have a great impact in biosphere [2–4]. Their importance and applications in the agriculture field and industries are well documented [5, 6].

A structure-activity relationship of organotin complexes has been explored using different carboxylate ligand [7–9]. The biological activity of the organotin compounds is probably due to the presence of easily hydrolysable groups yielding the intermediates, such as R_nSn_{4-n} (n = 2 or 3) moieties, which may bind with DNA [10], high as well as low affinity site of ATPase or haemoglobins [11, 12]. Although triorganotin compounds are commercially important as biocides, yet these also exhibit an interesting range of structural variations that may help to establish a structure-activity relationship [13]. The present work is the continuation of our interest in synthesis, characterization, and applications of organotin carboxylates [14–16]. We report here synthesis, characterization, and biological applications of organotin(IV) complexes of 4,5-dimethoxy-2-nitrobenzoic acid (**HL**).

[1] The article is published in the original.



EXPERIMENTAL

Materials and instrumentation. All chemicals used were of analytical grade. HL and organotin(IV) chlorides were purchased from Aldrich, while organotin oxides was from Alfa Aesar. These chemicals were used without further purification. The solvents were dried in situ according to a standard procedure [17]. All the reactions were carried out under argon atmosphere in dry toluene. Melting points were determined by using an Electro thermal based melting point apparatus model MP-D mitamura Riken Kogyo (Japan) and were uncorrected. IR spectra were recorded as KBr pallets on a Bio-Red Excaliber FT-IR, model 3000 MX Spectrum in the range of 4000–400 cm^{-1}. Mass data were recorded on a mass spectrometer model JMS 600H using the direct probe method and EI^+ as a source of ionization. NMR spectra were recorded on a Bruker 300 MHz spectrometer, using $CDCl_3$ as an internal reference. C, H, N analyses were performed with an organic elemental analyzer (model EA 1110, CE Instrument, Italy).

Synthesis of triorganotin carboxylates. HL (1 mmol) was dissolved in dry chloroform in a two-

Table 1. Elemental analysis data and some physical properties of HL and organotin(IV) compounds **I–VII**

Compound	M.p., °C	Yield, %	Molecular weight	Contents (calcd/found), %		
				C	H	N
HL	200		227			
I	58	78	390	36.92/36.87	4.35/4.41	3.58/3.62
II	185	80	516	48.83/48.93	6.78/6.70	2.71/2.79
III	168	80	576	56.25/56.33	3.99/3.89	2.43/2.33
IV	112	77	601	39.93/39.85	3.66/3.72	4.65/4.72
V	130	75	685	45.54/45.44	4.96/4.88	4.08/4.19
VI	170	72	725	49.65/49.73	3.58/3.64	3.86/3.97
VII	68	80	789	51.71/51.80	5.32/5.41	3.54/3.63

necked flask, and equimolar triethylamine (1 mmol) was added to it. The mixture was refluxed for 30 min. Then stoichiometric amount of triorganotin chloride was added to this solution and refluxed for 4–7 h. The reaction mixture was kept at room temperature overnight. The precipitated Et_3NHCl was then filtered off, and the solvent was evaporated under reduced pressure. The crude mass left behind was crystallized from an appropriate chloroform-*n*-hexane mixture (1 : 1).

$$R_3SnCl + HL + Et_3N \longrightarrow R_3SnL + Et_3NHCl,$$

R = Me (**I**), *n*-Bu (**II**), Ph (**III**).

Synthesis of diorganotin dicarboxylates. Stoichiometric amounts of carboxylic acid and organotin oxide were mixed in a single-neck flask in dry toluene and refluxed for 6–7 h in a round-bottom two-necked flask. Water formed during the reaction was continuously removed using a Dean-Stark apparatus. A yellow, transparent solution was obtained. Solvent was evaporated under reduced pressure by using a rotary evaporator. The mass left behind was crystallized from $CHCl_3$-acetone (1 : 1).

$$R_2S_nO + 2HL \longrightarrow R_2SnL_2 + H_2O,$$

R = Me (**IV**), *n*-Bu (**V**), Ph (**VI**), *n*-Oct (**VII**).

All the compounds **I–VII** were yellow in color, and obtained in more than 70% yield with melting point in the range of 58–185°C. These are fairly soluble in organic solvents like $CHCl_3$, DMSO, and THF. Physical data are given in Table 1.

RESULTS AND DISCUSSION

The IR spectra of complexes are compared with the spectrum of HL, and the important bands for structure assignment are given in Table 2. The complexation of ligand with tin is confirmed by the absence of a broad band at 3210 cm^{-1} due to ν(OH), thus showing the deprotonation of the carboxylic acid group. The difference between ν_{as}(COO) and ν_s(COO), i.e., Δν is important in the prediction of nature of the binding mode of the ligand and indicative of the coordination number around tin [18, 19].

As the value of difference Δν is less than 200 cm^{-1}, it suggests that the ligand behaves as bidentate toward the tin atom. The bands obtained in a range of 547–547–566 cm^{-1} are assigned to Sn–C bonds, whereas additional absorption in the range of 435–448 cm^{-1} shows the presence of Sn-O vibrations in these complexes.

The NMR signals are assigned by their intensity, multiplicity pattern, as well as their coupling constants and/or tin satellites. Their integration is in accordance with the composition expected for different fragments of the molecule.

The ^1H NMR spectra of the above reported complexes **I–VII** in a $CDCl_3$ solution show the absence of carboxylic proton, which was replaced by the organotin moiety. This information agrees well with what the IR data have revealed. All the protons in the reported com-

Table 2. Characteristic infrared bands of organotin(IV) compounds (cm^{-1})

Compound	ν_{as}(COO)	ν_s(COO)	Δν	ν(Sn–C)	ν(Sn–O)
HL	1500	1389	211		
I	1526	1379	147	563	440
II	1518	1360	158	547	446
III	1518	1375	143	564	448
IV	1528	1355	173	565	435
V	1528	1376	152	566	448
VI	1508	1363	145	563	445
VII	1522	1373	149	554	448

Table 3. ^{1}H NMR data of organotin carboxylates **I–VII***

Proton	Compound						
	I	**II**	**III**	**IV**	**V**	**VI**	**VII**
3	7.38 s.	7.42 s.	7.99 s.	8.72 s.	7.28 s.	7.41 s.	7.41 s.
6	7.28 s.	7.28 s.	7.98 s.	7.46 s.	7.23 s.	7.28 s.	7.27 s.
7	3.99 s.	3.17 s.	3.00 s.	3.19 s.	3.13 s.	3.95 s.	3.98 s.
8	3.97 s.	3.14 s.	2.98 s.	3.16 s.	3.10 s.	3.88 s.	3.96 s.
α	0.97 s. [58]	1.41–1.35 m.	7.70 m.	0.06 s. [77]	2.20–1.98 m.	7.39 m.	0.86–1.95 m.
β		1.25–1.30 m.	7.45 m.		1.75–1.80 m.	7.30 m.	
γ		0.78 (t., 7.0)	7.75 m.		1.00 (t., 7.0)	7.45 m.	

* Chemical shift in ppm, ^{n}J (^{1}H–^{1}H); ^{n}J (^{119}Sn–^{1}H) in Hz.

Table 4. ^{13}C NMR data of organotin carboxylates **I–VII***

Carbon atom	Compound						
	I	**II**	**III**	**IV**	**V**	**VI**	**VII**
1	124.4	125.7	127.3	127.9	126.9	120.2	119.3
2	141.4	140.5	143.7	148.9	143.5	143.0	139.1
3	110.9	110.5	112.2	110.5	111.7	111.3	104.4
4	152.0	152.4	151.5	152.5	151.7	152.1	149.6
5	149.5	149.2	150.9	151.9	151.2	150.9	147.8
6	112.7	106.6	127.4	126.7	119.9	106.9	108.4
7	56.53	56.6	55.1	56.5	55.5	56.7	54.1
8	56.51	56.5	55.0	56.4	55.2	56.4	54.0
9	170.5	176.5	173.1	174.0	175.3	179.4	176.0
α	14.02 [394]	29.6 [360]	138.5	29.6	29.3	129.9	37.2 [556]
β		27.4 [22]	137.4		27.7 [21]	127.5	31.2 [36]
γ		26.8 [63]	136.3		26.5	126.4	30.0
δ		14.5	130.7		14.6	125.3	29.3
α*							29.1
β*							26.7
γ*							22.4
δ*							14.1

* Chemical shift in ppm. Coupling ^{n}J (^{119}Sn–^{13}C) in Hz.

plexes have been identified at position and number with protons calculated from the incremental method [20]. ^{1}H NMR data are given in Table 3. The methyl protons of **I** appear as sharp singlet at 0.97 ppm with ^{n}J (^{119}Sn–^{1}H) coupling of 58.0 Hz. The methylene protons (CH_2) of *n*-octyltin(IV) moiety exhibit somewhat different behavior compared with the *n*-butyl groups of the respective complexes. The α-CH_2, β-CH_2, and γ-CH_2 to γ'-CH_2 protons give broad/multiplet signals at 0.86–1.95 ppm, which are consistent with the values calculated by the incremental method [20].

The aromatic carbon resonances are assigned by the comparison of experimental chemical shift with those calculated from the incremental method [20] or compared with literature data [8].

The carboxylate carbon shifts to the lower-field region in all complexes **I–VII** indicating participation of the carbonyl group COO in coordination to tin(IV)

Table 5. Mass spectral data of organotin(IV) compounds **I–VII**

Fragment/ion	**I**	**II**	**III**	**IV**	**V**	**VI**	**VII**
	m/z (%)						
R_2SnL		460(8)	500(100)		460(8)		572(8)
SnL	302(100)	302(12)	302(25)	302(4)	302(100)	302(13)	302(70)
R_3Sn^+	165(12)	291(8)	348(18)				
R_2Sn^+	150(17)	234(10)	272(8)	150(12)	234(9)	272(11)	346(8)
RSn^+	135(38)	177(8)	196(82)	135(28)	177(12)	196(12)	233(4)
Sn^+	120(8)	120(80)	120(65)	120(12)	120(10)	120(52)	120(7)
$C_9H_8NO_6^+$	226(65)	226(82)	226(15)	226(12)	226(38)	226(100)	226(73)
$C_7H_2^+$	86(12)	86(100)	86(32)	86(100)	86(2)	86(67)	86(32)
$C_4H_9^+$	75(60)	57(50)	57(3)	57(14)	57(50)	57(45)	75(100)
$C_4H_{11}O_2NSn^+$	211(9)	211(8)	211(8)	211(8)	211(8)	211(8)	211(8)
$C_9H_8O_4^+$	180(21)	180(27)	180(6)	180(24)	180(8)	180(21)	180(18)
$C_7H_2O_2^+$	124(41)	124(3)	124(10)	124(51)	124(19)	124(45)	124(36)
$C_6H_5O^+$	93(16)	93(32)	93(12)	93(26)	93(11)	93(16)	93(18)

[21]. The coupling constants ^nJ (^{119}Sn, ^{13}C) are important parameters for the structural characterization of organotin(IV) compounds and data is given in Table 4. For triorganotin(IV) compounds, the magnitude of ^1J (^{119}Sn, ^{13}C) coupling suggests the tetrahedral geometry around the tin atom in solution [22, 23]. As far as geometry of the diorganotin dicarboxylates in non-coordinating solvents is concerned, it is not defined with certainty due to the fluxional behavior of the carboxylate oxygen in their coordination with the tin atom [24]. However, the earlier reports suggest geometry in between penta- and hexacoordination [25, 26].

Mass spectrometry was carried out using electron beam of 70 eV. The major fragments along with their *m/z* and relative abundances are given in Table 5. The mass fragmentation pattern follows our previously established route [27]. In triorganotin and diorganotin derivatives no molecular ion peak is observed. The fragment ions are in good agreement with the expected structure of the compounds.

The complexes were checked for antifungal activity against different plant pathogens by using the Agar tube dilution protocol [28], and the data collected are listed in Table 6. Generally, all derivatives show markedly higher antifungal activity than the ligand with few exceptions. The triorganotin(IV) derivatives are found to be more active than the diorganotin derivatives, and the behavior is quite consistent with earlier report [29].

The activity varies with variation of the R groups attached to Sn. Apparently, the function of the ligand is to support the transport of the active organotin moiety to the site of action, where it is released by hydrolysis [30].

The synthesized complexes were tested for their antibacterial activity, using the agar well diffusion method [28]. The ligand and complexes **I–IV**, **VI**, **VII** were found to be inactive against all bacteria except complex **I**, **II**, **V**, and **VII** versus bacteria *Psendomonas*

Table 6. Antifungal data of organotin(IV) compounds*

Name of fungus	HL	**I**	**II**	**III**
Trichphyton longifusus	0	80	80	80
Candida albicans	0	0	0	90
Aspergillus flavus	45	90	40	0
Microsporum canis	0	80	80	70
Fusarium solani	0	90	0	75
Candida glabrata	0	0	0	90

* Complexes **IV–VII** shows no antifungal activity (except complex **VI** versus fungus Fusarium solani (40)).

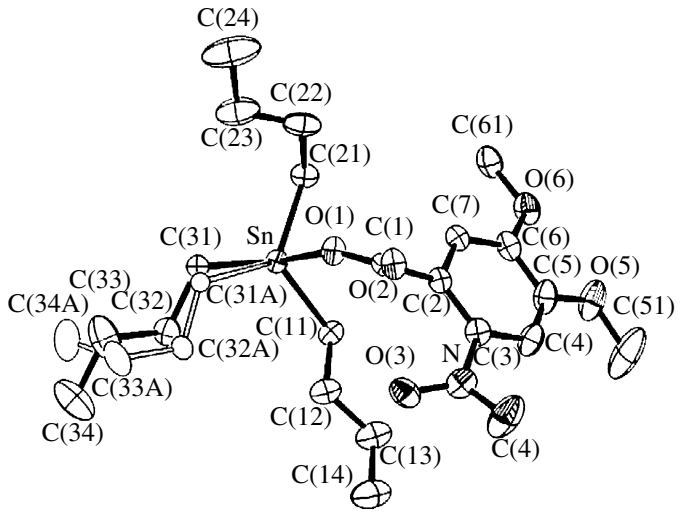


Fig. 1. ORTEP drawing of the X-ray structure of compound **II**.

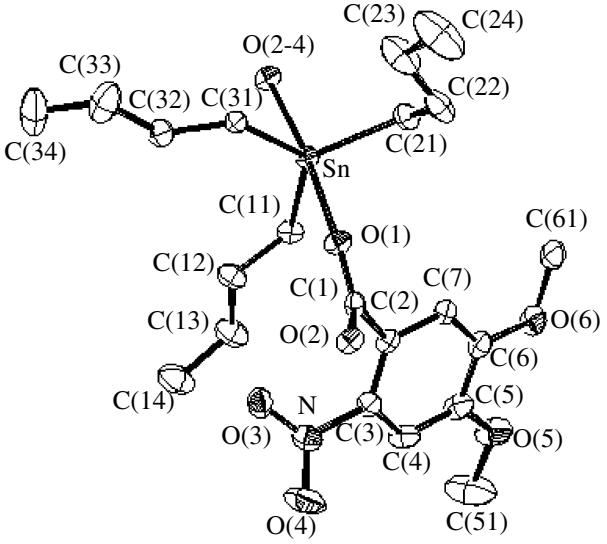


Fig. 2. Refined ORTEP drawing of the X-ray structure of compound **II**.

aeruginosa (13, 12, 15, and 15, respectively). Complex **V** is active against all bacteria, for example *Eschericha coli*, *Bacillus subtilis*, *Shigella flexenari*, *Staphylococcus aureus*, *Pseudomonas aeruginosa*, and *Salmonella typhi* (13, 15, 15, 15, 15 and 14, respectively). In the present study, the triorganotin(IV) derivatives are found to be more active than the diorganotin(IV) derivatives. This situation is in sharp contrast to the earlier assessment that an increase in the number of R groups on the tin(IV) atom enhances its biological activity. This anomalous behavior can be explained on the basis of the anionic ligand group that generally plays a secondary role.

The complexes were also screened for cytotoxicity data, using the Brine-shrimp (Artemia salina) bioassay lethality method [28]. The data show that ligand and compounds **I–III** and **V–VIII** shows no cytotoxicity. However, compound **IV** shows and LD_{50} value of 0.0255 μg/ml and exhibits significant toxicity.

The molecular structure of complex **II** is shown in Fig. 1. The complexes exhibit a carboxylate-bridged motif in which the Sn center shows *trans*-Bu_3SnO_2 trig-

Table 7. Crystal data and structure refinement for compound **II**

Parameter	Value
Empirical formula	$C_{21}H_{35}NO_6Sn$
Formula weight	516.19
Temperature, K	150(2)
Wavelength, Å	0.71073
Crystal system	Monoclinic
Space group	$P2_1/a$
Unit cell dimensions:	
a, Å	10.6065(2)
b, Å	18.9480(4)
c, Å	12.1146(3)
β, deg	94.7700(10)
V, Å^3	2426.26(9)
Z	4
ρ_{calcd}, mg m^{-3}	1.413
Absorption coefficient, mm^{-1}	1.086
F(000)	1064
Crystal size, mm	0.40 × 0.25 × 0.18
θ range for data collection, deg	3.26 to 27.48
Index ranges	$-13 \le h \le 12$ $-24 \le k \le 24$ $-15 \le l \le 1$
Reflections collected	30439
Independent reflections	5535 (R_{int} = 0.0670)
Reflections observed ($I > 2\sigma(I)$)	4297
Data completeness	0.997
Absorption correction	Semiempirical from equivalents
Max. and min. transmission	0.8285 and 0.6705
Refinement method	Full-matrix least-squares on F^2
Refined parameters	291
Goodness-of-fit on F^2	1.044
Final R indices ($I > 2\sigma(I)$)	R_1 = 0.0330 wR_2 = 0.0676
R indices (all data)	R_1 = 0.0531 wR_2 = 0.0748

Table 8. Selected bond lengths and bond angles for compound **II**

Bond	d, Å	Bond	d, Å
Sn–C(31)	2.133(7)	C(31)–C(32)	1.580(15)
Sn–C(11)	2.137(3)	C(32)–C(33)	1.47(3)
Sn–O(2)	2.2031(17)	C(33)–C(34)	1.52(2)
Sn–O(1)	2.4158(17)	C(31*A*)–C(32*A*)	1.481(14)
C(11)–C(12)	1.518(4)	C(32*A*)–C(33*A*)	1.56(2)
C(12)–C(13)	1.535(4)	C(33*A*)–C(34*A*)	1.480(17)
C(13)–C(14)	1.514(5)	O(1)–C(1)	1.250(3)
C(21)–C(22)	1.527(4)	O(2)–C(1)	1.268(3)
C(22)–C(23)	1.517(6)	O(2)–Sn	2.2031(17)
C(23)–C(24)	1.532(5)		
Angle	**ω, deg**	**Angle**	**ω, deg**
C(31)SnC(11)	126.2(2)	C(32)C(33)C(34)	114.8(17)
C(31)SnC(21)	116.3(2)	C(3)C(2)C(7)	116.9(2)
C(11)SnC(21)	117.30(11)	C(3)C(2)C(1)	124.5(2)
C(31)SnC(31*A*)	9.4(3)	C(7)C(2)C(1)	118.4(2)
C(11)SnC(31*A*)	116.7(2)	O(3)NO(4)	123.9(3)
C(21)SnC(31*A*)	125.6(2)	O(3)NC(3)	118.8(2)
C(31)SnO(2)	85.32(19)	O(4)NC(3)	117.3(3)
C(31)SnO(1)	84.74(19)	C(4)C(3)C(2)	122.8(3)
C(11)SnO(1)	90.63(9)	C(4)C(3)N	116.7(3)
C(21)SnO(1)	89.70(9)	O(6)C(6)C(5)	115.2(3)
C(31A)SnO(1)	84.31(18)	C(7)C(6)C(5)	119.9(3)
O(2)SnO(1)	170.02(6)	C(6)O(6)C(61)	117.5(2)
C(32)C(31)Sn	111.2(6)	C(6)C(7)C(2)	121.6(3)
C(33)C(32)C(31)	114.3(12)		

onal-bipyramidal coordination, the equatorial plane being defined by three R groups, while axial positions are occupied by two oxygen atoms. According to literature [31], the geometry around Sn atom can be characterized by $\tau = (\beta - \alpha)/60$, where β is the largest of the basal angles around the Sn atom. The angle values $\alpha = \beta = 180°$ correspond to a square-pyramidal geometry, and the value $\alpha = 120°$ corresponds to a perfect trigonal-bipyramidal geometry. Thus, the x value is equal to zero for a perfect square-pyramidal and one for perfect trigonal-bipyramidal. The calculated τ value for complex **II** is 0.079. This value indicates a slighly distorted trigonal-bipyramidal arrangement around the Sn atom. The angles of axial bonds (OSnO) are 170.02°, while the sum of the equatorial CSnC angles is 359.8°. The bond lengths are in agreement with the reported triorganotin(IV) carboxylates [32]. The intermolecular C=O ⟶ Sn coordination in compound **II** leads to infinite zigzag chains containing the Sn centers and carboxylate groups. The crystal data for **II** are listed in Table 7, selected bond lengths and bond angles are given in Table 8. There is disorder at the α-carbon of one butyl group (C(31) and C(31*A*)) in 1 : 1. Due to their close special proximity, they have only been refined isotropically. The refined structure is given in Fig. 2.

The atomic coordinates and other parameters of compound **II** have been deposited with the Cambridge Crystallographic Data Center (no. 699389; deposit@ccdc. cam.ac.uk).

Thus, 4,5-dimethoxy-2-nitrobenzoic acid in the reaction with organotin oxides/halides yields the organotin carboxylates in anhydrous toluene, which have been characterized by various analytical and spectro-

scopic techniques. The resulting triorganotin carboxylates are one-dimensional polymer as solid with trigonal bipyramidal geometry, while in solution, they are tetrahedral. Diorganotin carboxylates are octahedral in the solid phase, while in non-coordinating solvent the coordination number is between 5 and 6. Biological activity data show that all complexes exhibit the significant activity with few exceptions.

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