Turk J Chem
28 (2004) , 75 – 85.

^{1}H, ^{13}C, ^{119}Sn NMR, Mass, Mössbauer and Biological Studies of Tri-,Di- and Chlorodiorganotin(IV) Carboxylates

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Received 05.06.2003

The reaction of 2-[(2,3-dimethylphenyl)amino]benzoic acid (HL) with tri- and diorganotin(IV) chlorides yielded complexes of the type R_3SnL and R_2SnL_2 (R = *n*-C_4H_9, C_6H_5, $C_6H_5CH_2$, *n*-C_8H_{17}). The compounds R_2SnClL were obtained by a redistribution reaction between R_2SnL_2 and R_2SnCl_2. All the compounds were characterized by CHN analysis, infrared, multinuclear NMR, mass and Mössbauer spectroscopy. The possible mode of carboxylate group bonding in solid as well as in solution is proposed on the basis of infrared, Mössbauer and NMR (^{1}H, ^{13}C, ^{119}Sn) spectroscopy.

Introduction

It is well documented that organotin compounds have a wide range of industrial and biocidal applications because of their antifungal properties [1]. Various studies have shown that the replacement of a ligand (L) in such systems (R_3SnL, R_2SnL_2 and R_2SnClL) changes the toxicity effect of the organotin moiety[2,3].

In the course of our continuing investigation of organotin(IV) carboxylates, i.e., synthesis and characterization[4–7], X-ray crystal analysis[8–10] and biological[11–14] and industrial[15] applications, we report here a series of organotin(IV) derivatives of the biologically active ligand 2-[(2,3-dimethylphenyl)amino]benzoic acid (Figure 1) and some previous data for comparison[4]. These compounds are characterized by various instrumental techniques such as infrared, ^{1}H, ^{13}C and ^{119}Sn NMR, ^{119m}Sn Mössbauer and mass spectrometry.

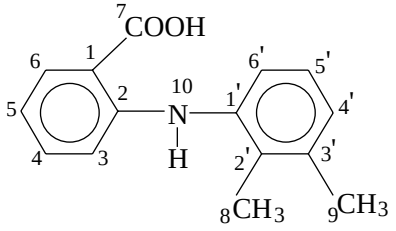

Figure 1. 2-[(2,3-Dimethylphenyl)amino]benzoic acid (HL).

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Results and Discussion

The tri- and diorganotin carboxylates were prepared by the reaction of 2-[(2,3-dimethylphenyl)amino] benzoic acid (HL) with the respective organotin chlorides in the presence of triethylamine [Eq (1)].

$$R_{4-n}SnCl_n + nEt_3N + nHL \rightarrow R_{4-n}SnL_n + nEt_3N.HCl \quad (1)$$

$$R = n-C_4H_9, C_6H_5CH_2, n-C_8H_{17} \text{ and } n = 1 \text{ or } 2$$

The chlorodiorganotin(IV) carboxylates were obtained by a redistribution reaction [Eq (2)].

$$R_2SnL_2 + R_2SnCl_2 \rightarrow 2R_2Sn(Cl)L \quad (2)$$

$$R = n-C_4H_9, C_6H_5, C_6H_5CH_2$$

All of the investigated compounds are solids and soluble in common organic solvents. Physical data for the series of compounds are reported in Table 1.

Table 1. Physical data[a,b] of organotin derivatives of 2-[(2,3-dimethylphenyl)amino]benzoic acid.

No.	Compounds (Empirical formula) Formula weight	M.p. (°C)	Yield (%)	% C calcd. (found)	% H calcd. (found)	% N calcd. (found)
(1)	*n*-Bu_3SnL ($C_{27}H_{41}NO_2Sn$) 530	135	70	61.13 (61.05)	7.73 (7.68)	2.64 (2.65)
(2)	*n*-Bu_2SnL_2 ($C_{38}H_{46}N_2O_4Sn$) 713	99	65	63.96 (63.90)	6.45 (6.40)	3.93 (3.90)
(3)	*n*-Bu_2SnClL ($C_{23}H_{32}NO_2ClSn$) 508.5	40	92	54.28 (53.88)	6.29 (6.41)	2.75 (2.73)
(4)	Ph_2SnClL ($C_{27}H_{24}NO_2ClSn$) 548.5	90	96	59.07 (58.80)	4.38 (4.18)	2.55 (2.49)
(5)	$Benz_3SnL$ ($C_{36}H_{35}NO_2Sn$) 632	65	85	68.35 (68.23)	5.54 (5.57)	2.22 (2.16)
(6)	$Benz_2SnL_2$ ($C_{44}H_{42}N_2O_4Sn$) 781	80	70	67.61 (67.50)	5.38 (5.41)	3.58 (3.60)
(7)	$Benz_2SnClL$ ($C_{29}H_{28}NO_2ClSn$) 576.5	110	96	60.36 (60.06)	4.84 (4.92)	2.42 (2.46)

[a] *n*-Bu = *n*-C_4H_9; Ph = C_6H_5; Benz = $C_6H_5CH_2$; L = $C_{15}H_{14}O_2N$

[b] Compounds **(1)** and **(2)** from reference 4.

Infrared spectroscopy

The infrared spectra of the compounds have been recorded as KBr or CsI disks in the range of 4000-250 cm^{-1}. The characteristic bands as given in Table 2 are ν(COO) (symmetric and asymmetric), ν(Sn-C), ν(Sn-O) and ν(Sn-Cl). The COO stretching vibrations are important to predict the bonding mode of the ligand. According to Lebl et al. the values of $\Delta\nu$ [$\Delta\nu = \nu_{asym.}$(COO) - $\nu_{sym.}$(COO)] can be divided into 3 groups[16]; (a) In compounds where $\Delta\nu$(COO) > 350 cm^{-1}, the carboxylate group binds in a monodentate fashion. However, other very weak intra- and intermolecular interactions cannot be excluded. (b) When $\Delta\nu$(COO) < 200 cm^{-1}, the carboxylate groups of these compounds can be considered to be bidentate. (c) In compounds where $\Delta\nu$(COO) > 200 cm^{-1} and < 350 cm^{-1} an intermediate state between monodentate and bidentate (anisobidentate) occurs. It has also been suggested that the $\Delta\nu$(COO) value in the chelating mode is less than the $\Delta\nu$(COO) in a bridging mode.[17]

Table 2. Infrared data[a] (cm^{-1}) for organotin derivatives of 2-[(2,3-dimethylphenyl)amino]benzoic acid.

No.	Compounds	ν (COO)		$\Delta\nu$	ν (Sn-C)	ν (Sn-O)	ν Sn-Cl
		asym	sym				
(1)	n-Bu_3SnL	1632 s	1440 s	192	550 w	450 m	-
(2)	n-Bu_2SnL_2	1630 s	1442 s	188	555 m	450 w	-
(3)	n-Bu_2SnClL	1605 s	1425 s	180	550 m	450 w	380 m
(4)	Ph_2SnClL	1570 s	1384 s	186	520 m	444 m	372 m
(5)	$Benz_3SnL$	1590 s	1370 s	220	540 w	465 m	-
(6)	$Benz_2SnL_2$	1578 s	1380 s	198	522 w	453 w	-
(7)	$Benz_2SnClL$	1571 s	1384 s	187	576 m	451 m	335 m
(8)	Oct_2SnL_2	1507 s	1325 s	182	525 m	429 m	-
	Ligand	1592 s	1340 s	252	-	-	-

[a]s, strong; w, weak; m, medium

From the preceding discussion it is proposed that in the investigated compounds, R_2SnL_2 and $R_2Sn(Cl)L$ have chelating-type carboxylates while the compounds R_3SnL have bridging carboxylate groups.

The bands for ν(Sn-C) and ν(Sn-O) are assigned in the range of 522-576 and 429-465 cm^{-1}, respectively. In chlorodiorganotin(IV) carboxylates, the ν(Sn-Cl) band is observed in the region 335-380 cm^{-1}.

Mass spectrometry

The mass fragmentation data are given in Tables 3-5. Molecular ion peaks of very low intensity are observed for almost all the compounds. In general, in triorganotin derivatives, the major fragmentation is the loss of an R group followed by the elimination of CO_2. An alternative route of fragmentation is the loss of the ligand anion first, followed by the loss of successive R groups until Sn^+ is obtained. For diorganotin dicarboxylates, the main fragmentation is due to the loss of 1 ligand which follows the loss of CO_2 and then successive loss of 2 R groups. Chloro derivatives follow the same pattern as the triorganotin carboxylates. When chloride is present, fragmentation by cleavage of R and L groups from Sn is again the predominant decomposition path. The first loss is due to an R group followed by CO_2 and then another R group.

NMR spectroscopy

^{1}H NMR data for tri-, di- and chlorodiorganotin(IV) derivatives are reported in Table 6. The expected resonances were assigned on the basis of their intensity and multiplicity pattern as well as their coupling constants. Comparing the spectra with the precursors, it is observed that most of the proton signals of the ligand are unchanged in the organotin derivatives, except for the N-H signal. The downfield shift of the N-H signal form δ 9.11 (in the free ligand) to δ 9.3-9.7 (in Sn-L complexes) is probably due to a transfer of charge towards the carboxylate group, which bonds to the more electropositive Sn. The $^{2}J[^{1}H,^{119}Sn]$ values for various compounds indicate that the compounds R_3SnL have a 4-coordinated environment while R_2SnL_2 and R_2SnClL show higher coordination numbers, probably 5, in non-coordinating solvents.

In the ^{13}C NMR spectra, resonances were assigned by comparing the experimental chemical shifts with those calculated by the incremental method[18] and literature values[19–23]. Almost all of the carbons are unchanged after complexation with Sn except the carboxylic carbons (COO) and C(1) which are shifted downfield by 4-6 ppm (Table 7). The observed shifts in both of these carbons are probably due to the

Table 3. Mass spectral data[a] for triorganotin 2-[(2,3-dimethylphenyl)amino]benzoates.

Fragmentations	**(1)**	% Int.	**(5)**	% Int.
$[(CH_3)_2C_6H_3NHC_6H_4CO_2SnR_3]^+$	531	10	633	25
$[(CH_3)_2C_6H_3NHC_6H_4CO_2SnR_2]^+$	474	44	542	14
$[(CH_3)_2C_6H_3NHC_6H_4SnR_2]^+$	430	43	498	30
$[(CH_3)_2C_6H_3NHC_6H_4SnR]^+$	373	28	407	32
$[(CH_3)_2C_6H_3NHC_6H_4Sn]^+$	316	100	316	100
$[(CH_3)_2C_6H_3NHC_6H_4COOH]^+$	241	38	241	41
$[(CH_3)_2C_6H_3NHC_6H_4]^+$	196	48	196	8
$[SnH]^+$	121	42	121	21
$[Sn]^+$	120	40	120	9
$[C_6H_3NH]^+$	90	18	90	28
$[C_5H_5]^+$	65	39	65	68

[a]Int. = Intensity

Table 4. Mass spectral data for diorganotin 2-[(2,3-dimethylphenyl) amino]benzoates.

Fragmentation ion	**(2)**	% Int.	**(6)**	% Int.	**(8)**	% Int.
$[[(CH_3)_2C_6H_3NHC_6H_4COO]_2SnR_2]^+$	714	13	782	18	825	15
$[[(CH_3)_2C_6H_3NHC_6H_4COO]_2SnR]^+$	657	10	691	12	712	20
$[(CH_3)_2C_6H_3NHC_6H_4COOSnR_2]^+$	474	100	542	22	585	100
$[(CH_3)_2C_6H_3NHC_6H_4COOSnR]^+$	417	45	451	30	472	5
$[(CH_3)_2C_6H_3NHC_6H_4Sn]^+$	316	58	316	100	316	50
$[(CH_3)_2C_6H_3NHC_6H_4COOH]^+$	241	32	241	40	241	60
$[(CH_3)_2C_6H_3NHC_6H_4]^+$	196	42	196	55	196	10
$[SnH]^+$	121	41	121	44	121	15
$[Sn]^+$	120	38	120	13	120	12
$[C_5H_5]^+$	65	42	65	70	65	10

[a]Int. = Intensity

Table 5. Mass spectral data for chlorodiorganotin 2-[(2,3-dimethylphenyl)amino]benzoates.

Fragmentations ion	**(3)**	% Int.	**(4)**	% Int.	**(7)**	% Int.
$[(CH_3)_2C_6H_3NHC_6H_4COOSn\ (Cl)R_2]^+$	510	22	550	5	578	n.o.[b]
$[(CH_3)_2C_6H_3NHC_6H_4COOSnR_2]^+$	474	100	514	100	542	100
$[(CH_3)_2C_6H_3NHC_6H_4SnR_2]^+$	430	54	470	32	498	52
$[(CH_3)_2C_6H_3NHC_6H_4SnR]^+$	373	32	393	21	407	33
$[(CH_3)_2C_6H_3NHC_6H_4Sn]^+$	316	24	316	50	316	70
$[R_2Sn]^+$	234	14	274	5	302	15
$[(CH_3)_2C_6H_3NHC_6H_4]^+$	196	8	196	80	196	13
$[SnH]^+$	121	51	121	14	121	48
$[Sn]^+$	120	32	120	6	120	10
$[C_5H_5]^+$	65	21	65	16	65	68

[a]Int. = Intensity,[b]not observed

transfer of charge from COO towards Sn and from N towards COO, which is evident from the ^{1}H NMR spectra too where the N-H signal shows a downfield shift. From the coupling constants $^{1}J[^{13}C,^{119}Sn]$, it is concluded that the triorganotin(IV) derivatives have a tetrahedral environment whereas the di- and chlorodiorganotin(IV) derivatives are either penta or hexa-coordinated. Using various equations[24,25], C-Sn-C bond angles were calculated for some of the compounds, which also confirms 4- and 5- coordination of the Sn in these compounds (Table 8).

The chemical shift δ^{119}Sn of organotin compounds covers a range of over 600 ppm and is quoted relative to tetramethyltin with downfield shifts from the reference compound with a positive sign. As the electron-releasing power of the alkyl group increases, the Sn atom becomes progressively more shielded and the δ^{119}Sn value moves to a higher field. These values are also dependent on the nature of X in $R_{4-n}SnX_n$ and generally move to a lower field as the electronegativity of the latter increases. One very important property of the ^{119}Sn chemical shift is that an increase in the coordination number of the Sn atom from 4 to 5, 6 or 7 usually produces a large upfield shift of δ^{119}Sn[26]. The ^{119}Sn NMR data for the investigated compounds fall in different coordination ranges, *i.e.*, 4, 5 or 6[27] (Table 9). The R_3SnL, compounds **(1), (2)** and **(5)** are clearly in the 4-coordination range while R_2SnClL, compounds **(3)**, **(4)** and **(7)** are in the 5-coordinating environment. The R_2SnL_2 compounds **(6)** and **(8)** are 6 coordinate.

Mössbauer spectroscopy

^{119m}Sn Mössbauer spectroscopy is a powerful tool for investigating the stereochemistry and bonding of organotin compounds. The 2 most important parameters are isomer shifts IS (mm s^{-1}) and quadrupole splittings QS (mm s^{-1}).

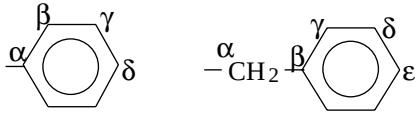
Isomer shift values are dependent upon the s-electron density at the Sn atom and should vary with Sn-ligand bonds. It is reported[26] that higher electronegativity of the ligand lowers the IS value. If the ligand remains constant, the isomer shift increases with the electron donating power of the alkyl group. In the present series it is observed that chloro derivatives have a lower IS value, probably due to the electronegativity of the Cl^{-} ion in the system. It is also observed that diorganotin compounds possessing 2 ligands have lower IS values than triorganotin compounds. A change in coordination number or stereochemistry in the Sn atom will also affect the isomer shift parameter, i.e., the higher the coordination number of Sn, the lower the isomer shift value. As seen from the data reported in Table 9, the tribenzyltin(IV) derivative has the highest IS value, indicating a tetra-coordinate Sn. This is further supported by the $\Delta\nu$ value from infrared data (Table 2). The IS values for diorganotin and chlorodiorganotin derivatives suggest *trans*-$R_2SnL_2$and *trans*-R_2SnClL structures, respectively.

Quadrupole splitting parameters are helpful in the elucidation of the stereochemistry of organotins. From the data reported in Table 9, it is concluded that Bu_3SnL has a *trans*- penta coordinated structure with 2 Sn-O axial bonds with different bond lengths. For diorganotin(IV) derivatives the QS value is more towards higher coordination with *trans*- geometry. According to literature data[26], the QS value for 5 coordinated, trigonal- bipyramidal chlorodiorganotin complexes ranges from 3.00 to 3.50 mms^{-1}. In the present series (Table 9), it is concluded on the basis of QS values that R_2SnClL compounds form 5-coordinated trigonal-bipyramidal geometry.

Table 6. ^{1}H NMR data^{a-c} for di-, tri- and chlorodiorganotin derivatives of 2-[(2,3-dimethylphenyl)amino]benzoate.

Proton	Bu_3SnL	Bu_2SnL_2	Bu_2SnClL	Ph_2SnClL	$Benz_3SnL$	$Benz_2SnL_2$	$Benz_2SnClL$	Oct_2SnL_2
3	6.8 d (9.4)	6.9 d (9.3)	6.9 d (9.3)	6.8 d (9.4)	6.85 d (9.4)	6.83 d (9.4)	6.79 d (9.4)	6.76 d (9.3)
4	7.3 t (7.9)	7.3 t (7.0)	7.32 t (7.7)	7.2 t (7.9)	7.3 t (7.9)	7.25 t (7.9)	7.29 t (7.7)	7.17 t (7.5)
5	6.7 t (8.1)	6.7 t (8.20)	6.71 t (8.2)	6.68 t (8.1)	6.71 t (8.0)	6.7 t (8.1)	6.8 t (8.2)	6.9 t (8.3)
6	8.2 d (8.0)	8.2 d (7.6)	8.06 d (8.1)	8.1 d (7.7)	8.2 d (7.6)	8.12 d (7.9)	8.2 d (7.9)	8.4 d (7.8)
8	2.3 s	2.2 s	2.21 s	2.2 s	2.3 s	2.3 s	2.4 s	2.24 s
9	2.5 s	2.3 s	2.35 s	2.3 s	2.35 s	2.32 s	2.34 s	2.38 s
10	9.7 s	9.3 s	9.31 s	9.5 s	9.4 s	9.5 s	9.6 s	9.26 s
$4'$	6.9 d (8.5)	7.0 d (9.3)	7.1 d (9.2)	6.8 d (8.9)	6.81 d (8.9)	7.21 d (9.1)	7.23 d (9.2)	7.08 d (9.2)
$5'$	7.1 t (7.5)	7.1 t (7.6)	7.15 t (7.6)	7.12 t (7.5)	7.0 t (7.5)	7.1 t (7.5)	7.12 t (7.5)	7.17 t (7.5)
$6'$	7.3 t (7.8)	7.3 t (6.8)	7.31 t (6.8)	7.2 t (7.9)	7.2 t	7.25 t	7.21 t	7.28 t
α	1.9-1.8 m	1.7-1.8 m	1.6-2.1 ^{2}J[69.0]	–	2.75 ^{2}J[62.4]	2.41 ^{2}J[68.1]	2.48 ^{2}J[68.1]	1.87-0.89 m
β	1.6-1.5 m	1.54-1.45 m	1.2-1.39 m	7.98 m	7.8 m	7.91 m	7.89 m	-
γ	1.6-1.5 m	1.54-1.45 m	–	7.62 m	7.3 m	7.2 m	7.00 m	-
δ	1.1 t (7.2)	0.96 t (7.0)	0.90 t (7.0)	7.57 m	7.5 m	7.52 m	7.45 m	-
ε	-	-	-	-	-	7.55 m	7.52 m	-

[a] Chemical shift in ppm(δ), $^{3}J(^{1}\text{H-}^{1}\text{H})$ in Hz, $^{2}J[^{119/117}\text{Sn-}^{1}\text{H}]$ in Hz.
[b] Multiplicity is given by: s = singlet, d = doublet, t = triplet, m = multiplet.
[c] α, β, γ, δ, ε are R groups



$-^{\alpha}CH_3$
$-^{\alpha}CH_2{}^{\beta}CH_2{}^{\gamma}CH_2{}^{\delta}CH_3$
$-^{\alpha}CH_2{}^{\beta}CH_2{}^{\gamma}CH_2{}^{\delta}CH_2{}^{\alpha'}CH_2{}^{\beta'}CH_2{}^{\gamma'}CH_2{}^{\delta'}CH_3$

Table 7. ^{13}C NMR data[a,b] for di-, tri- and chlorodiorganotin derivatives of 2-[(2,3-dimethylphenyl)amino]benzoate.

Carbon	Bu_3SnL	Bu_2SnL_2	Bu_2SnClL	Ph_2SnClL	$Benz_3SnL$	$Benz_2SnL_2$	$Benz_2SnClL$	Oct_2SnL_2		
1	113.2	111.3	112.9	113.1	114.1	114.2	113.9	113.6		
2	148.8	149.3	149.4	149.4	150.4	151.2	151.3	149.3		
3	113.2	113.4	113.5	113.5	112.9	113.3	113.6	113.4		
4	133.2	134.5	134.5	134.2	135.0	136.0	135.9	134.6		
5	115.8	116.2	116.1	116.0	116.3	117.2	116.9	116.2		
6	132.9	133.3	133.7	133.4	133.8	133.6	133.7	133.4		
7	178.5	177.4	176.8	177.5	176.9	178.8	177.3	177.5		
8	13.8	13.9	13.9	13.6	13.5	13.6	13.7	13.9		
9	20.5	20.7	20.6	20.6	20.9	21.1	21.2	20.6		
1′	139.3	138.9	138.8	140.0	139.9	140.2	140.1	139.0		
2′	131.7	131.9	132.0	132.2	132.3	132.5	132.6	131.9		
3′	137.7	138.1	138.1	138.4	138.8	139.1	139.2	138.2		
4′	122.4	122.6	122.6	122.9	122.3	121.6	122.1	122.5		
5′	125.6	125.8	124.9	126.0	125.9	125.7	125.8	125.9		
6′	126.0	126.4	126.4	126.4	126.6	126.8	126.7	126.4		
α	16.6 ^{1}J[352.6]	25.9 ^{1}J[589]	25.8	139.0 ^{1}J[650.0]	22.4 ^{1}J[263,271]	26.3 ^{1}J[316]	25.2 [310.3]	13.9	α'	33.3
β	27.7 ^{2}J[21.26]	26.9 ^{2}J[19.7]	27.2	136.6 ^{2}J[50.0]	139.1	134.6	137.7	29.7	β'	22.7
γ	26.9 ^{3}J[64.7]	26.6 ^{3}J[98.73]	26.1	129.0 ^{3}J[170.0]	129.5	129.9	129.2	29.1	γ'	20.6
δ		13.6	12.5	130.2 ^{4}J[13.6]	128.7	129.0	128.9	31.8	δ'	14.2
ε	-	-	-	-	126.5	127.0	127.3	-		

[a]Chemical shift in ppm(δ), $^{n}J[^{119/117}\text{Sn-}^{13}\text{C}]$ in Hz.
[b]For α, β, γ, δ, ε see footnote to Table 6

Table 8. C-Sn-C angles based on NMR parameters.

Compound	^{1}J[^{119}Sn-C]	θ (°)
Bu_3SnL	352.6	114.6
Bu_2SnL_2	589.0	136.6
Ph_2SnClL	650.0	116.3

Table 9. ^{119m}Sn Mössbauer data and ^{119}Sn NMR for organotin derivatives of 2-[(2,3-Dimethylphenyl)amino]benzoic acid.

No.	Compounds	IS	QS	Γ_1	Γ_2	QS/IS (ρ)	δ^{119} Sn (ppm)
(1)	Bu_3SnL	1.36	3.47	0.92	0.94	2.55	106.5
(2)	Bu_2SnL_2	1.31	3.25	1.02	0.99	2.48	-142
(3)	Bu_2SnClL	1.28	3.35	0.91	0.96	2.61	-125
(4)	Ph_2SnClL	1.21	3.29	0.94	0.94	2.719	-160
(5)	$Benz_3SnL$	1.53	3.46	0.99	0.94	2.26	-37
(6)	$Benz_2SnL_2$	1.32	3.30	0.89	0.88	2.50	-200
(7)	$Benz_2SnClL$	1.33	3.21	0.98	0.96	2.41	-105
(8)	Oct_2SnL_2	–	–	–	–	–	-201.4

Biological activity

Results of toxicity data and antibacterial and antifungal activity are reported in Table 10. Brine shrimps were used to check the toxicity of organotin(IV) carboxylates. Compounds (**5**), (**6**) and (**8**) do not show any positive lethality because according to previous reports[29,30] the toxicity of organotin compounds depends upon the nature or organic group. These compounds were also tested for their antibacterial activity by using 6 different bacteria. All the compounds show significant antibacterial activity against the tested bacteria, except against *Salmonella typhi* which show low activity. The tube diffusion method[31] shows that reported compounds are more active against *Trichophyton longiusus* while they do not show any antifungal activity against other fungi.

Experimental

All of the di- and triorganotin chlorides except for the benzyl derivatives were procured from aldrich or fluka and the tri- and dibenzyltin chlorides were prepared by the reported method[25]. All of the solvents were dried before use by the literature method[26]. The ligand, 2-[(2,3-dimethylphenyl)amino]benzoic acid, was kindly supplied by Eros, Geofman and Lahore Pharmaceuticals (Pvt) Ltd., as a pure powder, which was crystallized in dichloromethane before use.

Instrumentation

Melting point, were determined in a capillary tube using a model MP-D Mitamura Rikero Kogyo (Japan) electrothermal melting point apparatus. Infrared absorption spectra were recorded as KBr/CsBr pellets on a spectrum 1000 Perkin Elmer FT.IR spectrometer. The NMR spectra were recorded on a Bruker 250ARX spectrometer using $CDCl_3$ as an internal reference [δ^1H ($CDCl_3$) = 7.23: δ^{13}C ($CDCl_3$) = 77.0] ^{119}Sn NMR spectra were obtained with Me_4Sn [Ξ = 37.296665 MHz] as an external reference. Mössbauer spectra

Table 10. Toxicity data, antibacterial and antifungal activity for organotin(IV) derivatives of 2-[(2,3-dimethylphenyl)amino]benzoil acid.

Comp. No.	Toxicity Data				Antibacterial Activity						Antifungal Activity					
					Bacterium						Fungus					
	Dose (µg/mL)	No. of Shrimps	No. of Survivors	LD_{50} (µg/mL)	*Escherichia coli*	*Bacillus subtilis*	*Shigella flexenari*	*Stephyloco aureus*	*Pseudomonas aeruginosa*	*Salmoneila typhi*	*Trichophyton longiusus*	*Candida albicans*	*Aspergilla flavis*	*Microsporum canis*	*Fusarium solani*	*Candida geaberata*
(**5**)	100	30	10	−	10	17	10	12	11	−	89.4	0	0	60	0	0
	10	30	10													
	1	30	10													
(**6**)	100	30	10	−	10	10	10	10	12	−	42.1	0	0	0	0	0
	10	30	10													
	1	30	10													
(**8**)	100	30	10	−	10	−	−	10	11	−	57.8	0	0	0	0	0
	10	30	10													
	1	30	10													

were obtained with a constant acceleration microprocessor-controlled spectrometer (Cryoscopic Ltd., Oxford U.K.). A barium stannate source was used at room temperature and samples were packed in perspex disks and cooled to 80 K. Isomer shift data are relative to SnO_2.

Synthesis

The general method for the synthesis of tri- and diorganotin (IV) derivatives is the following: a quantity of 0.01 mol (2.41g) of 2-[(2,3-dimetheylphenyl)amino]benzoic acid (HL) was refluxed with 0.01 mol of Et_3N for 15 min in chloroform. Then 0.01 mol of triorganotin chloride or 0.005 mole diorganotin dichloride was added in solid form at room temperature and the mixture was further refluxed for 3-4 h under N atmosphere. After cooling the reaction mixture to room temperature, Et_3NHCl was filtered and the solvent was removed by a rotary evaporator. The solid mass left as residue was recrystalized from dichloromethane/hexan (1:1). The chloroderivatives were prepared by the method reported earlier[28].

Acknowledgements

S. Ali is grateful to Quaid-i-Azam University for financial support from the University Research Fund and the Alexander von Humboldt Foundation for a Georg Forster Research Fellowship. Prof. B. Wrackmeyer from the University of Bayreuth, Germany, is acknowledged for providing spectral facilities. Special thanks to Eros, Geofman and Lahore Pharmaceuticals (Pvt) Ltd, for providing 2-[(2,3-dimethylphenyl)amino]benzoic acid.

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