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Diorganotin complexes of carboxylates: synthesis and characterization

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Diorganotin complexes of monoisopropyl and monomethyl nadiate, succinate, and phthalate were synthesized and characterized by elemental analysis, FT-IR, ^{1}H NMR, ^{13}C NMR, and ^{119}Sn NMR spectroscopic techniques. The spectroscopic investigation demonstrated that carboxylate is bidentate in the diorganotin complexes. On the basis of $^{1}J(^{119}\text{Sn}–^{13}\text{C})$ and $^{2}J(^{119}\text{Sn}–^{1}\text{H})$ values, C–Sn–C bond angles were also calculated. The newly synthesized complexes were also screened for their antibacterial activities against Gram-positive and Gram-negative pathogenic strains of bacteria.

Keywords: Synthesis; Characterization; Organotin compounds

1. Introduction

Commercial significance of organometallic complexes containing lead, tin, and mercury has risen considerably [1]. Organotin complexes can be powerful fungicides and bactericides, depending on the constituent organic group and ligands [2–5].

Tetraorganotin complexes, tin tetrahalides, and stannanes (SnR_nH_{4-n}, where $n = 1–3$) are of synthetic value for production of complexes that have industrial value [6]. Industrial use of less toxic organotin complexes (R_2SnX_2 and $RSnX_3$) accounts for almost two-thirds of the total world's consumption of tin. Several organotin complexes such as di(*n*-butyl) tin laurate have been used as hydrochloric acid scavengers in PVC. Use of their complexes as selective biocides, insecticides, and pesticides has increased rapidly in recent years. Tributyl and triphenyltin chlorides find extensive use as biocidal additives in marine antifouling paints [6]. Synthesis and structural studies of neutral tin complexes derived from ligands with strategically placed donors (O, N, and S) have received attention [7–11].

Organotin carboxylates have received particular attention owing to their remarkable structural diversity and biological importance, for example, as pesticidal, antibacterial, antitumor agents, and wood preservatives [12–18]. Thus, the synthesis of organotin complexes

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having carboxylates has been a research focus [19–22]. Existing literature also proved that the biological activities of organotin carboxylates greatly depend on their structures, which are markedly related to properties of the carboxylic acid ligands and organic substituents bonded to tin.

In this article, we synthesized a series of organotin complexes using carboxylates and studied their characterization and antibacterial activities against Gram-negative and Gram-positive bacteria.

2. Experimental

2.1. *Materials*

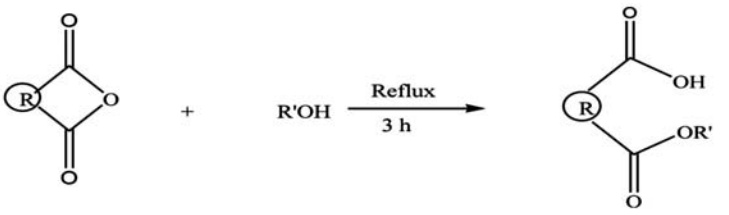
Manipulations of air and moisture-sensitive complexes were performed under nitrogen using standard Schlenk techniques. Diorganotin dichlorides, R_2SnCl_2 (R = Me, Bu), nadic anhydride (NA), succinic anhydride (SA), and phthalic anhydride (PA) all were obtained commercially from Sigma–Aldrich and used as received. Methanol and isopropanol (Spectrochem) were dried over sodium overnight followed by distillation. Triethyl amine (Et_3N) dried over KOH pellets for 24 h, chloroform (spectrochem) dried over calcium chloride for 72 h, hexane (spectrochem) and dichloromethane (spectrochem) also dried over sodium were distilled before use according to standard methods [23].

2.2. *Measurements*

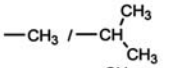
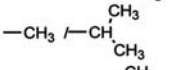
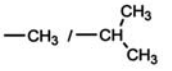
Structural characterization of ligands and diorganotin complexes was done using FT-IR, ^{1}H NMR, ^{13}C NMR, and ^{119}Sn NMR spectroscopic techniques. Elemental analysis was carried out using a EURO EA 3000 elemental analyzer. FT-IR spectra were recorded in KBr pellets from 4000 to 200 cm^{-1} using a Shimadzu FTIR 8700 spectrophotometer. ^{1}H NMR, ^{13}C NMR, and ^{119}Sn NMR spectra were recorded on a Bruker 300 MHz spectrometer using DMSO-d_6 as solvent. Tetramethylsilane was used as an internal standard for ^{1}H NMR, ^{13}C NMR spectra, and tetramethyltin as an internal standard for ^{119}Sn NMR spectra. FAB mass spectra in 3-nitrobenzyl alcohol (NBA) were recorded at room temperature on a JEOL SX 102/DA-6000 mass spectrometer/data system using argon/xenon (6 kV, 10 mA) as the FAB gas. The accelerating voltage was 10 kV.

2.3. *Synthesis*

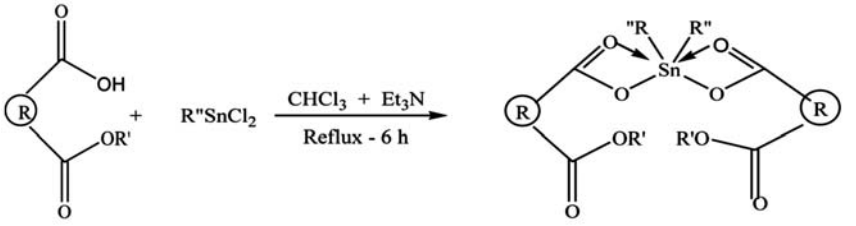
2.3.1. Synthesis of ligands. Different carboxylates (**L_1–L_6**) were prepared [24] using Scheme 1. A 10-fold excess of dry methanol (7.35 ml) and isopropanol (13.95 ml) were separately added to nadic anhydride, NA (3 g, 0.0182 mol) in a 250 ml round-bottom flask. The subsequent mixtures were heated under reflux for 3 h with constant stirring according to Scheme 1. Each reaction mixture was then evaporated under reduced pressure to remove excess of the respective alcohols, and the products were dried in a desiccator under reduced pressure to give 90–95% of compound. These two ligands were designated as NM and NIP, respectively.



where

R	R'	Sample designaton	FMW
		NM / NIP	194, 222
		SM / SIP	132, 160
		PM / PIP	180, 208

Scheme 1. Synthesis of ligands.



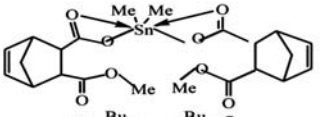
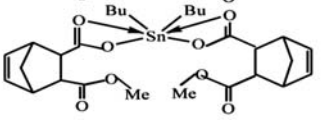
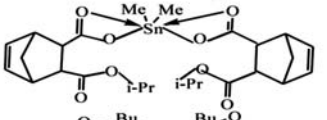
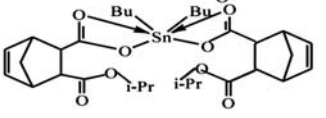
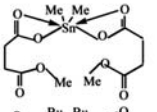
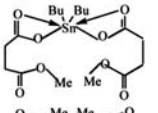
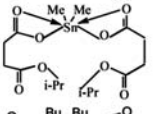
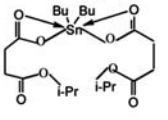
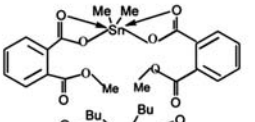
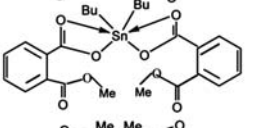
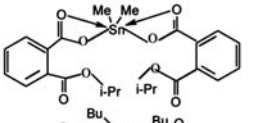
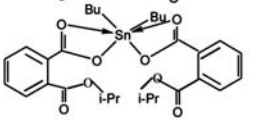
Scheme 2. Synthesis of diorganotin complexes.

Similarly, all other ligands were prepared by reacting succinic anhydride (SA) or phthalic anhydride (PA) with 10-fold excess of the dry methanol (M) and isopropanol (IP). The ligands thus obtained were designated as SM, SIP, PM, and PIP, respectively.

2.3.2. Synthesis of diorganotin(IV) complexes. Diorganotin complexes (**1**–**12**) were prepared by treating 2 mol of half-esters (ligands) with 1 mol of dialkyltin dichloride using Scheme 2.

A solution of NM (388 mg, 0.002 mol) in 10 ml chloroform was added to a mixture of dimethyltin dichloride (M) (219 mg, 0.001 mol) and Et_3N (0.3 ml). The resultant mixture was heated under reflux with stirring for 6 h (Scheme 2) and then allowed to stand at 25 °C for 10 h. The precipitated $Et_3NH^+Cl^-$ was filtered and the filtrate was evaporated under vacuum to yield a solid which was purified by recrystallization in a mixture of dichloromethane/hexane (1 : 1). It is designated as NMM.

Similarly, all other diorganotin(IV) complexes were prepared by reacting NM, NIP, SM, SIP, PM, and PIP with dimethyltin dichloride (M) and dibutyltin dichloride (B). Sample designation of diorganotin(IV) complexes is given in Scheme 3.

S.No.	Organotin Complexes	Sample Designation
1.		NMM
2.		NMB
3.		NIPM
4.		NIPB
5.		SMM
6.		SMB
7.		SIPM
8.		SIPB
9.		PMM
10.		PMB
11.		PIPM
12.		PIPB

where Me =-CH_3, Bu =-$CH_2CH_2CH_2CH_3$, i-Pr =-$CH(CH_3)_2$

Scheme 3. Sample designation of organotin(IV) complexes.

2.4. *Antibacterial studies*

Screening of the complexes for their antibacterial activities was performed by employing Broth Microdilution MIC method [25]. Using sterile microtitre plates, 0.2 ml of Mueller–Hinton Broth was added to each of the 96 wells. Doubling dilutions of each compound were made in the wells, thus, a plate contained 0.5–100 μg/ml dilutions of 12 different complexes and of ampicillin. In each plate, one well was kept as positive control (broth + inoculum) and another as negative control (broth only). The inoculum was adjusted to a turbidity equivalent to McFarland 0.5 turbidity standard. The inoculum was suitably diluted so as to get a final concentration of approximately 5×10^5 cfu/ml of bacteria in each well. Each well was inoculated with 0.01 ml of the prepared inoculum using a multichannel micropipette and the plates were incubated overnight at 37 °C. The MICs of these complexes and ampicillin were determined by using the standard protocol of NCCLS Broth Microdilution MIC method.

3. Results and discussion

3.1. *Chemistry*

Scheme 1 shows the synthesis of ligands. Dehydrochlorination occurred during the synthesis (Scheme 2). The designation of the compounds is shown in Scheme 3. The analytical data have been shown in table 1.

3.2. *IR spectra*

FT-IR spectra of all the complexes have been summarized in table 2. The absence of bands at 2500–3000 cm^{-1} due to O–H bond of the ligands and at 360 cm^{-1} due to Sn–Cl bond and presence of band at 650–675 cm^{-1} [26] due to Sn–O bond in all the organotin complexes indicated that dehydrochlorination had taken place leading to removal of proton of carboxyl and both Cl^- of Sn with formation of Sn–O bonds. The band at 450–475 cm^{-1} indicated formation of O→Sn [27]. The shifting of C=O band of carboxylic acid to lower

Table 1. Analytical data of complexes.

					Found (Calcd) (%)		
No.	Compound	Formula	FMW	Yield (%)	C	H	Sn
1	NMM	$C_{22}H_{28}O_8Sn$	539	80	48.9 (49.0)	5.1 (5.2)	22.1 (22.0)
2	NMB	$C_{28}H_{40}O_8Sn$	623	79	53.9 (53.9)	6.3 (6.5)	19.1 (19.0)
3	NIPM	$C_{26}H_{36}O_8Sn$	595	78	52.4 (52.5)	6.0 (6.1)	20.2 (20.0)
4	NIPB	$C_{32}H_{48}O_8Sn$	679	75	56.5 (56.6)	7.1 (7.1)	17.7 (17.5)
5	SMM	$C_{12}H_{16}O_8Sn$	407	77	35.3 (35.4)	3.9 (3.9)	29.3 (29.2)
6	SMB	$C_{18}H_{28}O_8Sn$	491	76	43.9 (44.0)	5.6 (5.7)	24.4 (24.2)
7	SIPM	$C_{16}H_{24}O_8Sn$	463	74	41.4 (41.5)	5.1 (5.2)	25.8 (25.6)
8	SIPB	$C_{22}H_{36}O_8Sn$	547	72	48.2 (48.3)	6.5 (6.6)	21.5 (21.7)
9	PMM	$C_{20}H_{20}O_8Sn$	507	74	47.2 (47.4)	3.9 (4.0)	23.6 (23.4)
10	PMB	$C_{26}H_{32}O_8Sn$	591	73	52.7 (52.8)	5.3 (5.4)	20.3 (20.1)
11	PIPM	$C_{24}H_{28}O_8Sn$	563	71	51.1 (51.2)	4.9 (5.0)	21.3 (21.1)
12	PIPB	$C_{30}H_{48}O_8Sn$	647	70	54.6 (54.9)	7.1 (7.4)	18.3 (18.1)

Table 2. FT-IR spectral data of complexes in cm^{-1}.

No.	Compound	ν_{asym}(COO)	ν_{sym}(COO)	$\Delta\nu$(COO)	ν(O→Sn)	ν(Sn–O)	ν_{asym}(Sn–C) ν_{sym}(Sn–C)
1	NMM	1583	1454	129	463	674	648, 552
2	NMB	1583	1452	131	463	674	647, 551
3	NIPM	1584	1454	130	464	675	648, 551
4	NIPB	1585	1454	131	463	674	648, 551
5	SMM	1583	1454	129	461	672	636, 552
6	SMB	1583	1456	127	463	674	634, 553
7	SIPM	1584	1454	130	460	671	636, 553
8	SIPB	1583	1454	130	461	672	651, 552
9	PMM	1584	1452	132	450	650	650, 555
10	PMB	1584	1454	129	455	656	650, 554
11	PIPM	1583	1453	130	455	655	651, 555
12	PIPB	1583	1454	129	453	654	650, 555

frequencies was seen, due to involvement of O in coordinate bond formation. However, bands due to ester (which is already present in the reactant) were observed at nearly the same positions both in ligands and complex showing that ester group was not involved in bond formation.

The bands due to ν_{asym}(COO) and ν_{sym}(COO) were observed between 1640–1575 cm^{-1} and 1420–1310 cm^{-1}, respectively (table 2). The difference between ν_{asym}(COO) and ν_{sym}(COO) was less than 200 cm^{-1}, indicating that the carboxylates were chelated and bidentate to metal [24,27].

3.3. *NMR spectra*

The ^{1}H NMR spectra of ligands showed a signal at δ 10.2 due to free –COOH group which disappeared in complexes, further supporting the dehydrochlorination reaction. Signals due to tin-alkyl protons in dibutyltin(IV) and dimethyltin(IV) complexes were observed at δ 0.8–1.7 [28,29]. In all diorganotin(IV) complexes, singlet due to $-OCH_3$ and multiplet due to protons of $-OCH(CH_3)_2$ group were observed at δ 3.6–3.7 and δ 4.6–4.7, respectively.

In **1**–**4**, multiplets of olefinic protons appeared at δ 5.6 and bridging methylene protons were at δ 1.8 ppm. Complexes **5**–**8** had multiplets of methylene protons at δ 2.5–2.6 ppm and in **9**–**12** the aromatic protons were observed at δ 7.6–8.2 ppm. Other ^{1}H NMR signals of complexes were observed at their usual positions and are summarized in table 3. The proton integrations were consistent with molecular formulas.

^{13}C NMR signals of carboxylic carbon of carboxylic acid group in all organotin(IV) complexes were observed downfield (δ 172.0–178.0) of their corresponding ligands (δ 167.6), suggesting bonding of ligands through both carboxylic oxygens of carboxylic acid to tin [30]. However, carbons of ester group were observed at nearly the same position (δ 180–182) in ligands and complexes, indicating non-involvement of ester in bonding. Carbons of alkyl groups that is, methyl and butyl attached to tin atom were observed at δ 9.4–9.6 and δ 13.6–27.4, respectively. Carbons of methyl and methine attached to oxygen of acid were observed at δ 50–51 and δ 68–70, respectively.

In **1**–**4**, olefinic carbons appeared at δ 135–137 and bridging carbons were observed at δ 43–44. Methylene carbons attached to carbonyl in **5**–**8** were at δ 28.7–34.0 and in **9**–**12** the aromatic protons were at δ 127.4–134.8. Other ^{13}C NMR signals of all complexes were observed at their usual positions and are summarized in table 4.

Table 3. ^{1}H NMR spectral data of complexes.

No.	Compound	δ (ppm) Sn–CH$_3$	Sn–^{1}CH$_2$–^{2}CH$_2$–^{3}CH$_2$–^{4}CH$_3$	CH–CH–COO–	–O–^{1}CH$_3$, –O–^{2}CH(^{3}CH$_3$)$_2$	Olefinic protons	Aromatic protons
1	NMM	1.0 (s) ^{2}J(^{119}Sn, ^{1}H) 69.3	–	2.3 m, 2.4 m	3.6 s (1)	5.6	–
2	NMB	–	0.9 t (4), 1.3–1.4 m (1, 2, 3)	2.3 m, 2.4 m	3.6 s (1)	5.6	–
3	NIPM	1.0(s) ^{2}J(^{119}Sn, ^{1}H) 70.2	–	2.4 m, 2.5 m	4.6 m (2), 1.4 d (3)	5.6	–
4	NIPB	–	0.9 t (4), 1.3–1.4 m (1, 2, 3)	2.3 m, 2.6 m	4.7 m (2), 1.4 d (3)	5.6	–
5	SMM	0.9(s) ^{2}J(^{119}Sn, ^{1}H) 72.2	–	2.6 m, 2.8 m	3.6 s (1)	–	–
6	SMB	–	0.8 t (4), 1.4–1.5 m (1, 2, 3)	2.4 m, 2.6 m	3.6 s (1)	–	–
7	SIPM	0.9(s) ^{2}J(^{119}Sn, ^{1}H) 70.3	–	2.4 m, 2.6 m	4.6 m (2), 1.3 d (3)	–	–
8	SIPB	–	0.8 t (4), 1.3–1.4 m (1, 2, 3)	2.5 m, 2.6 m	4.6 m (2), 1.3 d (3)	–	–
9	PMM	0.9(s) ^{2}J(^{119}Sn, ^{1}H) 75.2	–	–	3.7 s (1)	–	7.6–8.1 (4H, m)
10	PMB	–	0.9 t (4), 1.3–1.7 m (1, 2, 3)	–	3.7 s (1)	–	7.5–8.0 (4H, m)
11	PIPM	0.9(s) ^{2}J(^{119}Sn, ^{1}H) 78.5	–	–	4.6 m (2), 1.4 d (3)	–	7.6–8.2 (4H, m)
12	PIPB	–	0.9 t (4), 1.3–1.7 m (1, 2, 3)	–	4.6 m (2), 1.4 d (3)	–	7.6–8.1 (4H, m)

Table 4. ^{13}C NMR and ^{119}Sn NMR spectral data of complexes.

No.	Compound	–COO–	Sn–CH_3	Sn–$^1CH_2$–$^2CH_2$–$^3CH_2$–$^4CH_3$	CH–CH–COO–	–O–$^1CH_3$, –O–$^2CH(^3CH_3)_2$	Olefinic carbons	Aromatic carbons	^{119}Sn
1	NMM	177.1, 175.3	9.5	–	31.3, 36.1	51.0(1)	135.9, 135.9	–	−320.1
2	NMB	178.3, 175.2	–	25.9(1), 7.3(2), 26.10(3), 3.7(4) $^1J(^{119}Sn,^{13}C)$ 610.5 $^2J(^{119}Sn,^{13}C)$ 49.6 $^3J(^{119}Sn,^{13}C)$ 83.1	31.6, 35.6	50.7(1)	136.0, 136.0	–	−315.3
3	NIPM	178.4, 174.6	9.4	–	32.5, 36.2	69.0(2), 22.3(3)	136.8, 136.8	–	−330.0
4	NIPB	177.4, 174.2	–	25.3(1), 7.2(2), 26.1(3), 13.6(4) $^1J(^{119}Sn,^{13}C)$ 605.3 $^2J(^{119}Sn,^{13}C)$ 47.2 $^3J(^{119}Sn,^{13}C)$ 82.8	31.3, 36.2	69.2(2), 22.1(3)	137.0, 137.0	–	−290.5
5	SMM	174.3, 172.2	9.5	–	30.1, 29.8	51.01(1)	–	–	−300.0
6	SMB	174.1, 173.2	–	25.6(1),27.3(2), 26.2(3), 13.7(4) $^1J(^{119}Sn,^{13}C)$ 608.9 $^2J(^{119}Sn,^{13}C)$ 48.8 $^3J(^{119}Sn,^{13}C)$ 82.9	29.0, 28.7	51.01(1)	–	–	−325.2
7	SIPM	173.3, 172.1	9.5	–	30.0, 29.8	69.0(2), 22.3(3)	–	–	−313.0
8	SIPB	174.2, 173.1	–	25.9(1), 27.3(2), 26.10(3), 13.7(4) $^1J(^{119}Sn,^{13}C)$ 608.3 $^2J(^{119}Sn,^{13}C)$ 49.3 $^3J(^{119}Sn,^{13}C)$ 83.4	29.8, 29.8	69.0(2), 22.3(3)	–	–	−305.6
9	PMM	178.1, 174.3	9.4	–	–	51.0(1)	–	131.8(1), 132.1(2), 127.1(3), 134.1(4), 132.7(5), 131.8(6)	−337.2
10	PMB	178.2, 175.1	–	25.4(1), 27.4(2), 26.80(3), 13.7(4) $^1J(^{119}Sn,^{13}C)$ 608.7 $^2J(^{119}Sn,^{13}C)$ 48.8 $^3J(^{119}Sn,^{13}C)$ 83.3	–	51.0(1)	–	131.7(1), 132.3(2),127(3), 134.2(4), 132.7(5), 131.9(6)	−360.4
11	PIPM	177.3, 174.2	9.5	–	–	70.0(2), 23.1(3)	–	131.5(1), 131.9(2), 129.1 (3),133.8(4), 132.2(5), 131.5 (6)	−323.7
12	PIPB	177.1, 174.3	–	25.4(1), 27.4(2), 26.50(3), 13.7(4) $^1J(^{119}Sn,^{13}C)$ 610.8 $^2J(^{119}Sn,^{13}C)$ 49.9 $^3J(^{119}Sn,^{13}C)$ 83.4	–	70.0(2), 23.0(3)	–	131.8(1), 132.1(2), 128.1(3), 134.1(4), 132.4(5), 131.7(6)	−367.3

The presence of a singlet in the ^{119}Sn NMR spectra from δ −290 to δ −367 indicates six-coordinate tin in all the complexes [31–33]. The signals appeared much upfield due to large electron density on Sn due to oxygen donors of ligand. The appearance of singlets showed that there was only one isomer.

The C–Sn–C bond angles, θ were calculated using the following two equations [34,35] and have been summarized in table 5:

$$\theta = 0.0105|^2J(^{119}\mathrm{Sn} - ^{1}\mathrm{H})|^2 - 0.799|^2J(^{119}\mathrm{Sn} - ^{1}\mathrm{H})| + 122.4 \quad (1)$$

$$^{1}J(^{119}\mathrm{Sn} - ^{13}\mathrm{C}) = 9.99\theta - 746 \quad (2)$$

3.4. *Mass spectra*

Every effort to obtain crystals did not produce a single crystal. Evidence in support of the structures of these compounds came from FAB mass spectral studies.

Under the FAB mass condition, compounds displayed molecular ions at 539 (NMM), 623 (NMB), 595 (NIPM), 679 (NIPB), 407 (SMM), 491 (SMB), 463 (SIPM), 547 (SIPB), 507 (PMM), 591 (PMB), 563 (PIPM), and 647 (PIPB). Isotopic ions were observed corresponding to the different isotopes of tin. The observation of molecular ions in the mass spectra of the respective compounds confirmed monomers of the compounds. figures 1 and 2 show the mass spectra of PMM and NIPM, respectively.

3.5. *Antibacterial studies*

Antibacterial activities were evaluated using two Gram-positive (*Staphylococcus aureus*, *Staphylococcus epidermidis*) and two Gram-negative (*Escherichia coli*, *Pseudomonas aeruginosa*) bacteria (table 6). Against both the Gram-positive and Gram-negative bacteria, however, complexes SMB, SIPB, and PMB have the greatest effects over 24 h. This was due to the presence of electron-withdrawing group which lowers electron density in the compound. Comparison of electron density among various complexes could predict the

Table 5. Calculated C–Sn–C bond angles of complexes.

No.	Compound	$^{2}J(^{119}\mathrm{Sn},^{1}\mathrm{H})$	Bond angle	$^{1}J(^{119}\mathrm{Sn},^{13}\mathrm{C})$	Bond angle
1	NMM	69.3	127.3		
2	NMB			610.5	135.8
3	NIPM	70.2	126.7		
4	NIPB			605.3	135.3
5	SMM	72.2	125.3		
6	SMB			608.9	135.6
7	SIPM	70.3	126.7		
8	SIPB			608.3	135.6
9	PMM	75.2	123.1		
10	PMB			608.7	135.6
11	PIPM	78.5	120.4		
12	PIPB			610.8	135.8

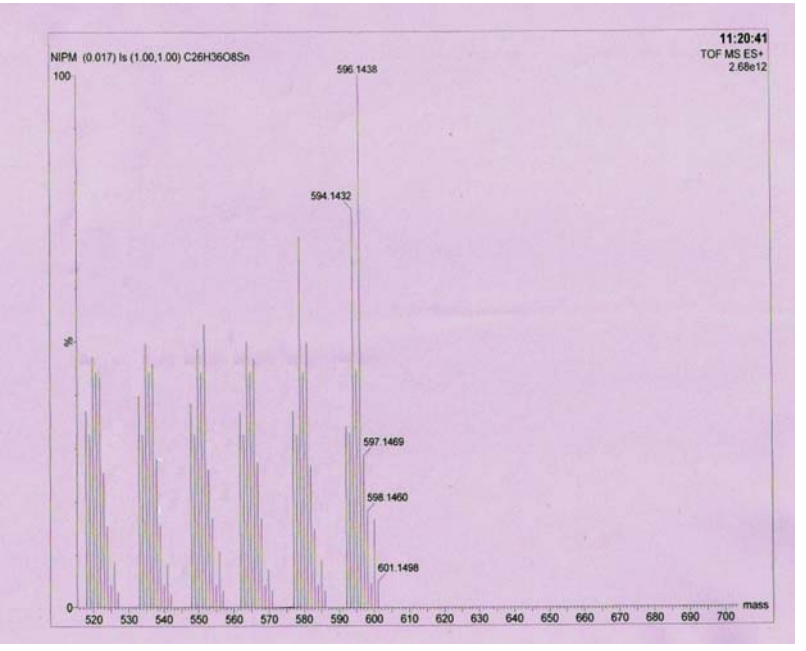


Figure 1. Mass spectra of NIPM.

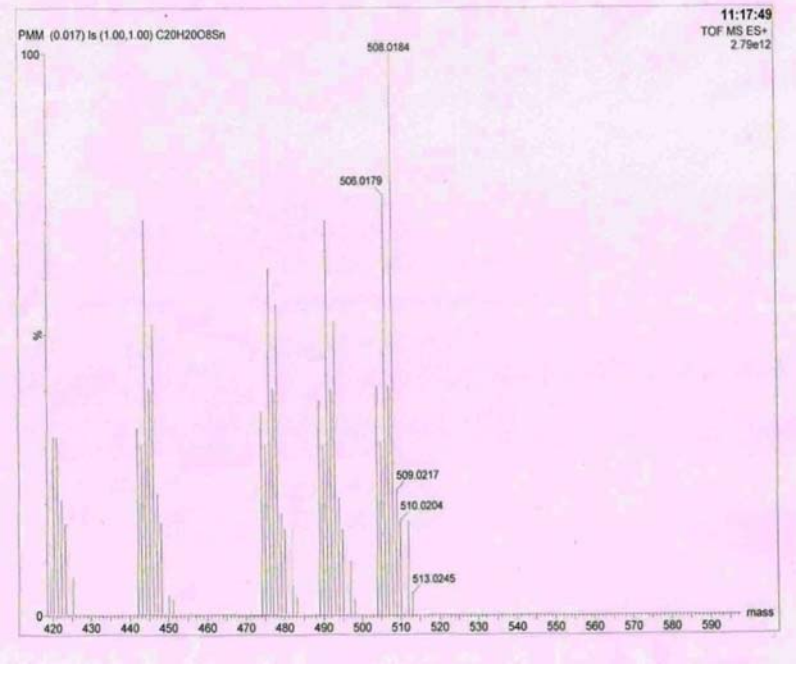


Figure 2. Mass spectra of PMM.

Table 6. Antibacterial activities of complexes by MIC method in $\mu g/mL^{-1}$.

No.	Compound	*P. aeruginosa*	*E. coli*	*S. aureus*	*S. epidermidis*
1	NMM	1000	250	1000	1000
2	NMB	250	131.2	125	125
3	NIPM	1000	500	1000	1000
4	NIPB	250	250	125	125
5	SMM	1000	500	1000	1000
6	SMB	31.2	31.2	31.2	31.2
7	SIPM	500	500	500	500
8	SIPB	31.2	31.2	31.2	125
9	PMM	1000	1000	500	1000
10	PMB	31.2	31.2	31.2	31.2
11	PIPM	1000	500	1000	250
12	PIPB	62.5	62.5	62.5	62.5

extent of interaction with the bio-receptor. The solvent used to prepare the stock solutions (DMSO) played no role in growth inhibition.

4. Conclusion

Organotin complexes studied here are six-coordinate. Organotin(IV) derivatives of dibutyl tin dichloride showed best MIC against *Escherichia coli*, *Staphylococcus aureus*, *Staphylococcus epidermidis,* and *Pseudomonas aeruginosa* compared to dimethyl tin dichloride derivatives.

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