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Synthesis, Characterization and Biochemical Activity of Homo and Heterobimetallic Derivatives of Dibutyltin Containing Mixed Chelating Ligands

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ABSTRACT

$Bu_2Sn(OPr^i)_2$'s interaction with Schiff base (L^aH), where $L^a = L^1 = OC_6H_4CH{=}NCNSC_6H_4$ (**1**) and $L^a = L^2 = O_2NC_6H_4CH{=}NCH_2CH_2O$ (**2**) yields the precursor derivatives $Bu_2Sn(L^a)(OPr^i)$, which on equimolar reaction with glycol (HOGOH), where G = G^1 = OCH(Me)CH(Me)O, and G = G^2 = $OC(Me)_2C(Me)_2O$, and diethanolamine $RN(CH_2CH_2OH)_2$ yield the derivative of the type $Bu_2Sn(L^a)(L^bH)$ [where $L^a = L^1$, $L^b = G^1$ (**3**); $L^a = L^1$, $L^b = G^2$ (**4**); $L^a = L^2$, $L^b = G^1$ (**5**); $L^a = L^2$, $L^b = G^2$ (**6**); $L^a = L^1$, $L^b = RN(CH_2CH_2O)_2$ where R=H (**7**); $L^a = L^1$, $L^b = RN(CH_2CH_2O)_2$ where R=CH_3 (**8**); $L^a = L^2$, $L^b = RN(CH_2CH_2O)_2$ where R=H (**9**); $L^a = L^2$, $L^b = RN(CH_2CH_2O)_2$ where R= CH_3 (**10**)]. Homometallic mixed ligand derivatives (**3**)-(**10**) on 1:1 reaction with aluminium isopropoxide form monomeric heterobimetallic mixed chelating ligand derivatives of type $[Bu_2Sn(L^a)(L^bAl(OPr^i)_2]$ (**11**)-(**18**) respectively. All these derivatives have been characterized by elemental analysis and spectroscopic [IR, NMR ^1H, ^{119}Sn, and ^{27}Al] studies. The synthesized derivatives were screened for their in vitro antimicrobial activity against gram +ve bacteria i.e. *S.aureus* and gram -ve bacteria i.e. *E.Coli*. All the derivatives showed enhanced antimicrobial activity than the present ligands.

1. Introduction

Due to the chelating property, world of chemistry have synthesized homometallic derivatives with Schiff base [1-5] and diol (glycols, diethanolamine) [6-11]. There is few work on heteroleptic derivative in which presence of glycols or diethanolamine chelating ligand with Schiff base is found [12-17].

During previous years, we have investigated metal complexes of mixed chelating ligand in our laboratory and synthesis of heterometallic heteroleptic metal complex of Bu_2Sn is given in this paper. Schiff base, glycol and diethanolamine has many applications because of azomethine, hydroxyl group and nitrogen or and hydroxyl group respectively. Schiff based containing metal derivatives have many application such as antibacterial, antifungal, anticancer, anti-inflammatory, anticonvulsant, and analgesic agents. Metal based ethanolamine derivatives use as buffers, catalysts, inhibitors, ion exchangers and dyes during last few years, now they are drawing attention for their biological role in bacterial and fungal metabolism [18-23]. Out of many application, biological activity is an important one.

This work synthesized and studied heteroleptic derivatives using spectroscopic and analytical techniques. Using the disc diffusion method, the antibacterial properties of all the derivatives were assessed against two different bacterial species. The ligand used for the synthesis of these derivatives are shown in Fig. 1.

2. Experimental Methods

Using oven-dried (130–140 °C) quick-fit glassware with interchangeable joints, all experiments were conducted under strictly anhydrous conditions. The glassware was cooled either in desiccators filled with fused $CaCl_2$ or by adequately shielding it from atmospheric moisture using guard tubes and side tubes fielded with self-indicating silica gel. Solvent such as benzene, n-hexane, isopropyl alcohol, ethyl alcohol and dichloromethane were purified and dried by standard procedures [24]. Diethanolamines $RN(CH_2CH_2OH)_2$ [R = H, Me] were dried and purified by refluxing over $Al(OPr^i)_3$, followed by distillation at 148 °C/1 mmHg (R=H) and 148 °C/0.3 mmHg (R=Me) respectively. 2,3-Butanediol and 2,3-dimethyl-2,3-butanediol were dried and purified by refluxing over $Al(OPr^i)_3$ followed by fractional distillation at 182 °C/758 mmHg and 171 °C/755 mmHg respectively.

Schiff base were prepared by equimolar condensation of HOC_6H_4CHO or $O_2NC_6H_4CHO$ with appropriate amine $C_6H_4CNSNH_2$ or $H_2NCH_2CH_2OH$ in benzene under refluxing conditions.

Aluminium and dibutyltin isoproxides were also made using techniques from the literature [25,26]. It was found that tin was an oxide and aluminium an oxinate [27]. Kjeldahl's method was used to calculate the percentage of nitrogen. The azeotrope's isopropyl alcohol content was calculated using the oxidimetric method. Cryoscopic measurements of molecular weights in benzene solution were made. Using TMS as an internal reference, ^1H (89.55/300.40 MHz) NMR data were obtained in $CDCl_3$ on JEOL 300AL NMR spectrometers. ^{27}Al (23.79/78.18 MHz) and ^{119}Sn (33.35/111.95 MHz) Tetra methyltin and an aqueous solution of aluminum nitrate were used as external references for the NMR spectra obtained in benzene, respectively.IR (4000-400 cm^{-1}) spectra were captured using a Nicolet Magna 550 spectrophotometer using Nujol mulls. A Perkin-Elmer series II CHNS/O analyser 2400 was used to record the analysis of carbon and hydrogen.

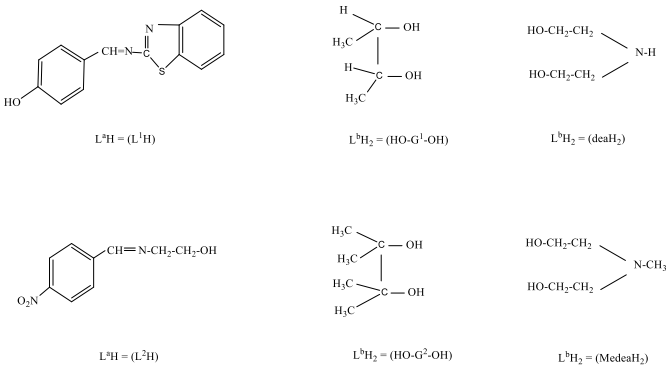

Fig. 1 Ligands that are employed in the creation of novel derivatives

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2.1 Synthesis of Homometallic Dibutyltin Derivatives

2.1.1 Synthesis of $Bu_2Sn(L^1)(OPr^i)$ (1)

A benzene solution of freshly prepared $Bu_2Sn(OPr^i)_2$ (8.90 g, 25.42 mmol) was added to a benzene (~30 mL) solution of $HOC_6H_4CH{=}NCNSC_6H_4$ (L^1H) (6.46 g, 25.42 mmol). The resultant brown color solution was refluxed under a fractionating column with continuous azeotropic distillation of the isopropyl alcohol for approximately five hours. During the aforementioned time, isopropyl alcohol was collected and its amount estimated. Refluxing was terminated once the reaction was complete, as shown by the absence of isopropyl alcohol in the distillation. Reduced pressure was used to extract the solvent (benzene) from the resultant solution, yielding a brown-coloured sticky solid (13.86 g, 82%).

Using the same process as for (**1**), the other derivatives (**2**)–(**10**) in the quantitative yields were also constructed. Table (2) contains a collection of analytical and physical details.

2.2 Synthesis of Heterobimetallic Dibutyltin Derivatives

Heterobimetallic dibutyltin derivatives have been prepared by the reaction of homometallic mixed ligand $Bu_2Sn(L)(O\text{-}G\text{-}OH)$ or $Bu_2Sn(L)(RdeaH)$ with aluminium alkoxide. The preparative details of only one typical derivative are provided here due to similarities in the synthetic procedure:

2.2.1 Synthesis of $Bu_2Sn(L^1)(O\text{-}G^1\text{-}O\text{-}Al(OPr^i)_2)$ (11)

The mixture of (**3**) (1.99 g, 3.46 mmol) and $Al(OPr^i)_3$ (0.76 g, 3.46 mmol) in benzene (~40 ml) was refluxed for approximately 4 hours while isopropyl alcohol was continuously removed azeotropically. Refluxing was stopped once the reaction was complete, and volatiles were extracted from the solution under reduced pressure to produce a yellow waxy solid. This solid, when recrystallized from a 1:2 mixture of n-hexane and dichloromethane at -20 °C, yielded 2.84 g (84%) of the analytically pure product (**11**).Compounds (**12**) through (**18**) were made using a method akin to (**11**). Details on analysis are provided in Table (2).

3. Results and Discussion

(1) Reaction of $Bu_2Sn(OPr^i)_2$ with Schiff bases (L^aH) in benzene yields homonuclear derivatives according the following equation:

$$Bu_2Sn(OPr^i)_2 + L^aH \xrightarrow[\text{reflux}]{\text{benzene}} Bu_2Sn(L^a)(OPr^i) + Pr^iOH\uparrow$$

L^aH = (L^1H) = $HOC_6H_4CH{=}NCNSC_6H_4$ and
(L^2H) = $O_2NC_6H_5CH{=}NCH_2CH_2OH$

Now this $Bu_2Sn(L^a)(OPr^i)$ react with glycols or diethanolamines.

Reaction of $Bu_2Sn(L^a)(OPr^i)$ with glycols (HO-G-OH) in benzene yields mixed ligand derivatives according the following Eq.(1)

$$Bu_2Sn(L^a)(OPr^i) + HO\text{-}G\text{-}OH \xrightarrow[\text{reflux}]{\text{benzene}} Bu_2Sn(L^a)(O\text{-}G\text{-}OH) + Pr^iOH\uparrow \quad \text{Eq.(1)}$$

HO-G-OH where G = $CH(CH_3)CH(CH_3)$ = (G^1) and
= $C(CH_3)_2C(CH_3)_2$ = (G^2)

Reaction of $Bu_2Sn(L^a)(OPr^i)$ with diethanolammine ($RdeaH_2$) in benzene yields mixed ligand derivatives according the following Eq.(2)

$$Bu_2Sn(L^a)(OPr^i) + R\text{-}deaH_2 \xrightarrow[\text{reflux}]{\text{benzene}} Bu_2Sn(L^a)(R\text{-}deaH) + Pr^iOH\uparrow \quad \text{Eq.(2)}$$

R-$deaH_2$ = H-$deaH_2$ = H-N (CH_2-CH_2-OH)$_2$ and
Me-$deaH_2$ = CH_3-N (CH_2-CH_2-OH)$_2$

Reaction of $Bu_2Sn(L^a)(O\text{-}G\text{-}OH)$ with $Al(OPr^i)_3$ in benzene yields heteronuclear derivatives according the following Eq.(3)

$$Bu_2Sn(L^a)(O\text{-}G\text{-}OH) + Al(OPr^i)_3 \xrightarrow[\text{reflux}]{\text{benzene}} Bu_2Sn(L^a)(O\text{-}G\text{-}O\text{-}Al(OPr^i)_2 + Pr^iOH\uparrow \quad \text{Eq.(3)}$$

Reaction of $Bu_2Sn(L^a)(R\text{-}deaH)$ with $Al(OPr^i)_3$ in benzene yields heteronuclear derivatives according the following Eq.(4),

$$Bu_2Sn(L^a)(R\text{-}deaH) + Al(OPr^i)_3 \xrightarrow[\text{reflux}]{\text{benzene}} Bu_2Sn(L^a)(R\text{-}dea\text{-}Al(OPr^i)_2 + Pr^iOH\uparrow$$

.... Eq. (4)

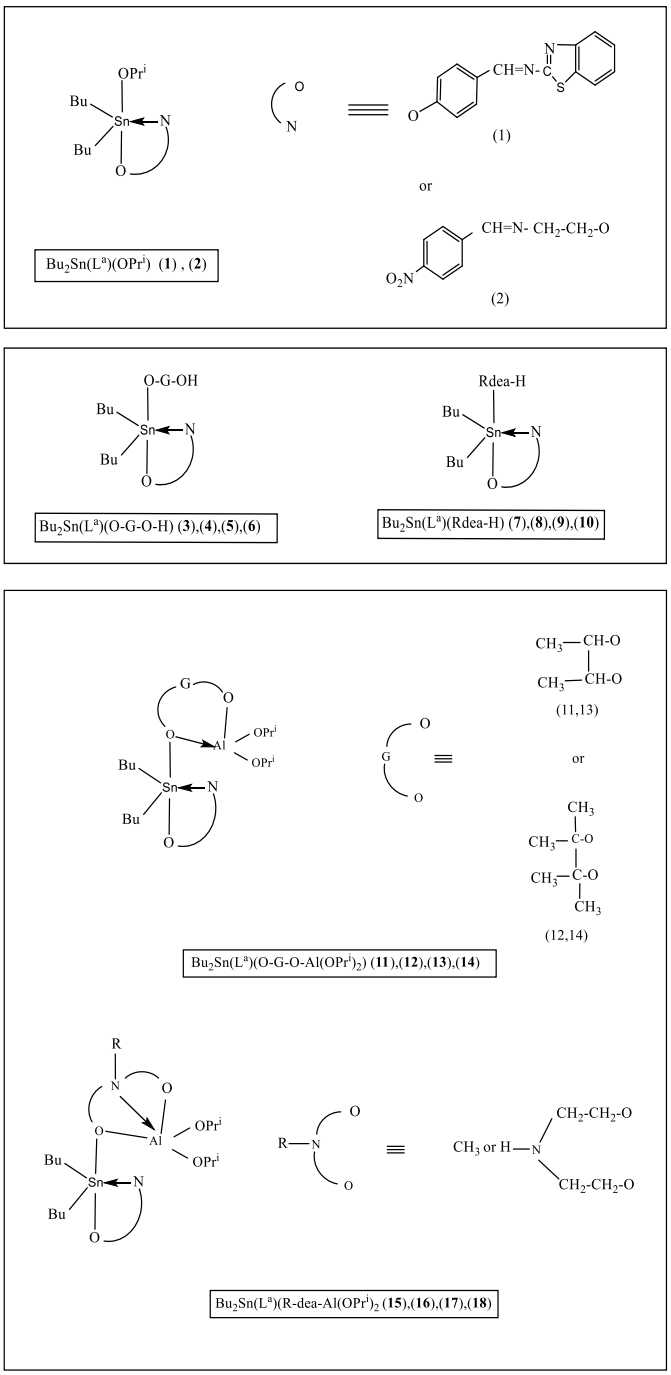

Fig. 2 Suggested structures for derivatives (**1**) - (**18**)

3.1 Spectroscopic Studies

3.1.1 Infrared Spectra

Every derivative's infrared spectrum was captured within the 4000-400 cm^{-1} region. The characteristic stretching vibration band of υ(OH) in complexes (**1**) and (**2**) was absent in the IR spectra, indicating that the band was present in the Schiff base ligand. The schiff base was bidentately coupled to the tin atom, according to the IR spectra of derivatives (**1**) - (**18**). The spectra of (**1**) - (**18**) show a shift in the vibration of the azomethine group to a lower frequency of ± 13 cm^{-1}. This shift indicates the manner of coordination between azomethine N and tin metal [1,28].

The regions with C-O stretching frequency are 1045±5 and 1235±5 cm^{-1}, respectively, because of the phenoxide and glycoxide groups. For derivatives (**1**) - (**18**), a smaller frequency shift of approximately 10 cm^{-1} in the phenolic υ(C-O) supports metal phenolate bonding through the phenolic oxygen. The emergence of new bonds in the ranges 480±5 and 450±6 cm^{-1}, respectively, has been attributed to υ(Al-O) and υ(Al←N) [9,14]. The heterometallic derivatives (**11**) - (**18**) exhibit absorption owing to glycol or diethanolamine and isopropoxide group in the anticipated locations (Table 1).

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3.1.2 NMR Spectra

The position of multiplates in the 6.99–8.23 ppm area caused by aromatic protons remains nearly unchanged, according to a comparison of the ^{1}H NMR spectrum data (Table-1) of the derivatives with the parent ligands. Additionally, the signals of the azomethine protons found in the 8.91–9.31 ppm region are protected, multiplates caused by 0.89–1.52 ppm of Bu_2Sn [1,28]. The remaining OH group in the derivatives (**3**)–(**6**) is responsible for the broad peak that appears at 1.19–3.18 ppm.

In the 1.15 - 3.75 ppm (CMe_2/CHMe) and 3.48 - 4.50 ppm (CHMe) areas [9,11], the signal characteristic of a glycollate moiety is found in the derivatives (**3**) - (**6**) and (**11**) - (**14**). Derivatives (**7**) - (**10**) and (**15**) - (**18**) show the signal for deprotanated diethanolamine moieties caused by NCH_2 and OCH_2 protons, respectively, in the areas of 2.59 to 3.84 ppm and 3.85 to 3.93 ppm. The zones corresponding to 1.18 - 1.32 ppm and 3.48 - 4.02 ppm show the signals attributed to the methyl and methine protons of isopropoxyl groups [9,11].

Table 1 List of significant spectrum data for the derivatives (**1**) - (**18**)

Derivatives	IR (cm^{-1})	NMR (δ, ppm)		
		^{1}H	^{119}Sn	^{27}Al
1	1625 υ(C=N), 1521 υ(C=N) thiol, 1132 υ(C-N), 752 υ(C-S), 1542 υ(C=C), 962 υ(C-H), 1235 υ(C-O) phenolic, 1045 υ(C-O) alcoholic, 542 υ(Sn-O), 581 υ(Sn-C), 519 υ(Sn-N), 1150,1130 υ(O-Pr^i).	0.90 (m, 6H, $Sn(CH_2)_3Me$), 1.23 (br, 18H, $Sn(CH_2)_3Me$ + CHMe), 4.01 (br, 1H, CHMe), 7.01-8.12 (m, 8H, aromatic-H), 9.31 (s, 1H, CH=N)	-142	-
2	1612 υ(C=N), 1544 υ(C=C), 964 υ(C-H), 1045 υ(C-O) alcoholic, 1370 υ(C-O) alcoholic(L), 543 υ(Sn-O), 582 υ(Sn-C), 521 υ (Sn-N),1152,1131 υ(OPr^i), 1424 υ(NO_2).	0.89 (m, 6H, $Sn(CH_2)_3Me$), 1.52 (m, 18H, $Sn(CH_2)_3Me$ + CHMe), 4.52 (m, 3H, NCH_2 +CHMe), 5.87(t, 2H, OCH_2), 7.96-8.01 (m, 4H, aromatic-H), 8.91 (s, 1H, CH=N)	-144	-
3	1644 υ(C=N), 1525 υ(C=N) thiol, 1130 υ(C-N), 754 υ(C-S), 1540 υ(C=C), 964 υ(C-H), 1238 υ(C-O) phenolic, 1046 υ(C-O) alcoholic, 544 υ(Sn-O), 583 υ(Sn-C), 521 υ(Sn-N).	0.91 (m, 6H, $Sn(CH_2)_3Me$), 1.15 (d, 6H, CHMe), 1.66(m, 12H, $Sn(CH_2)_3Me$), 3.18(br, 1H, OH), 3.75 (m, 2H, CHMe), 7.04-8.14 (m, 8H, aromatic-H), 9.29 (s, 1H, CH=N)	-180	-
4	1645 υ(C=N), 1528 υ(C=N) thiol, 1132 υ(C-N), 756 υ(C-S), 1542 υ(C=C), 962 υ(C-H), 1236 υ(C-O) phenolic, 1048 υ(C-O) alcoholic, 546 υ(Sn-O), 584 υ(Sn-C), 524 υ(Sn-N).	0.93 (m, 6H, $Sn(CH_2)_3Me$), 1.26 (s, 12H, CMe_2), 1.62 (m, 12H, $Sn(CH_2)_3Me$), 1.97 (br, 1H, OH), 6.99-8.01 (m, 8H, aromatic-H), 9.30 (s, 1H, CH=N)	-188	-
5	1622 υ(C=N), 1546 υ(C=C), 962 υ(C-H), 1049 υ(C-O) alcoholic, 1372 υ(C-O) alcoholic(L), 544 υ(Sn-O), 584 υ(Sn-C), 524 υ(Sn-N), 1422 υ(NO_2).	0.91 (m, 6H, $Sn(CH_2)_3Me$), 1.18 (d, 6H, $CHMe_2$), 1.58 (m, 12H, $Sn(CH_2)_3Me$), 3.16 (br, 1H, OH), 3.74 (m, 2H, CHMe),4.53 (t, 2H, NCH_2), 5.89 (t, 2H, OCH_2) 7.99-8.04 (m, 4H, aromatic-H), 8.94 (s, 1H, CH=N)	-158	-
6	1618 υ(C=N), 1544 υ(C=C), 962 υ(C-H), 1328 υ(CMe_2), 1046 υ(C O) alcoholic, 1375 υ(C-O) alcoholic(L), 546 υ(Sn-O), 582 υ(Sn-C), 522 υ(Sn-N), 1420 υ(NO_2).	0.94 (m, 6H, $Sn(CH_2)_3Me$), 1.39 (m, 24H, CMe_2 + $Sn(CH_2)_3Me$), 1.19 (br, 1H, OH), 4.53 (t, 2H, NCH_2), 5.88 (t, 2H, OCH_2), 7.95-8.08 (m, 4H, aromatic-H), 8.92 (s, 1H, CH=N)	-187	-
7	1646 υ(C=N), 1528 υ(C=N) thiol, 1134 υ(C-N), 752 υ(C-S), 1544 υ(C=C), 962 υ(C-H), 1236 υ(C-O) phenolic, 1046 υ(C-O) alcoholic, 546 υ(Sn-O), 584 υ(Sn-C), 522 υ(Sn-N), 2950, 2836 υ(CH_2) dea.	0.91 (m, 6H, $Sn(CH_2)_3Me$), 1.23 (m, 12H, $Sn(CH_2)_3Me$), 3.20 (t, 4H, NCH_2), 3.81 (t, 4H, OCH_2), 4.42 (s, 2H, NH + OH), 7.04-8.16 (m, 8H, aromatic-H), 9.30 (s, 1H, CH=N)	-184	-
8	1644 υ(C=N), 1526 υ(C=N) thiol, 1132 υ(C-N), 754 υ(C-S), 1540 υ(C=C), 964 υ(C-H), 1236 υ(C-O) phenolic, 1048 υ(C-O) alcoholic, 544 υ(Sn-O), 582 υ(Sn-C), 522 υ(Sn-N), 2952, 2838 υ(CH_2) dea.	0.90 (m, 6H, $Sn(CH_2)_3Me$), 1.21 (m, 12H, $Sn(CH_2)_3Me$), 2.31 (s, 3H,NMe), 2.59 (t, 4H, NCH_2), 3.86 (br, 5H, OCH_2 + OH), 7.06-8.19 (m, 8H, aromatic-H), 9.30 (s, 1H, CH=N)	-189	-
9	1615 υ(C=N), 1544 υ(C=C), 963 υ(C-H), 1048 υ(C-O) alcoholic, 1372 υ(C-O) alcoholic(L), 544 υ(Sn-O), 584 υ(Sn-C), 524 υ(Sn-N), 1424 υ(NO_2). 2954, 2835 υ(CH_2) dea.	0.94 (m, 6H, $Sn(CH_2)_3Me$), 1.24 (m, 12H, $Sn(CH_2)_3Me$), 3.24 (m, 6H, NCH_2 + NCH_2(dea)), 3.84 (m, 4H, OCH_2 (L) + OH(dea) + NH), 3.81 (t, 4H, OCH_2) 7.08-8.14 (m, 4H, aromatic-H), 9.19 (s, 1H, CH=N)	-182	-
10	1618 υ(C=N), 1546 υ(C=C), 962 υ(C-H), 1046 υ(C-O) alcoholic, 1374 υ(C-O) alcoholic(L), 546 υ(Sn-O), 582 υ(Sn-C), 520 υ(Sn-N), 1420 υ(NO_2). 2952, 2836 υ(CH_2) dea.	0.92 (m, 6H, $Sn(CH_2)_3Me$), 1.24 (m, 12H, $Sn(CH_2)_3Me$), 2.30 (s, 3H, NMe) 3.19 (m, 6H, NCH_2(L) + NCH_2(dea)), 3.81 (m, 7H, OCH_2 (L) + OCH_2(dea) + OH), 7.10-8.19 (m, 4H, aromatic-H), 9.20 (s, 1H, CH=N)	-174	-
11	1642 υ(C=N), 1532 υ(C=N) thiol, 1132 υ(C-N), 752 υ(C-S), 1542 υ(C=C), 962 υ(C-H), 1238 υ(C-O) phenolic, 1048 υ(C-O) alcoholic, 545 υ(Sn-O), 582 υ(Sn-C), 524 υ(Sn-N), 480 υ(Al-O), 1152,1130 υ(OPr^i).	0.90 (m, 6H, $Sn(CH_2)_3Me$), 1.20 (br, 18H, CHMe + $CHMe_2$), 1.67(br, 12H, $Sn(CH_2)_3Me$), 3.48 (br, 2H, CHMe), 4.03 (m, 2H, $CHMe_2$) 7.09-8.09 (m, 8H, aromatic-H), 9.31 (s, 1H, CH=N)	-184	52
12	1644 υ(C=N), 1530 υ(C=N) thiol, 1136 υ(C-N), 752 υ(C-S), 1544 υ(C=C), 964 υ(C-H), 1236 υ(C-O) phenolic, 1050 υ(C-O) alcoholic, 546 υ(Sn-O), 584 υ(Sn-C), 522 υ(Sn-N), 482 υ(Al-O), 1154,1131 υ(OPr^i).	0.90 (m, 6H, $Sn(CH_2)_3Me$), 1.19 (br, 24H, CMe_2 + $CHMe_2$), 1.61 (m, 12H, $Sn(CH_2)_3Me$), 4.02 (m, 2H, CHMe), 7.02-8.19 (m, 8H, aromatic-H), 9.21 (s, 1H, CH=N)	-174	58
13	1620 υ(C=N), 1542 υ(C=C), 960 υ(C-H), 1050 υ(C-O) alcoholic, 1374 υ(C-O) alcoholic(L), 546 υ(Sn-O), 586 υ(Sn-C), 522 υ(Sn-N), 484 υ(Al-O), 1152,1132 υ(OPr^i), 1419 υ(NO_2).	0.92 (m, 6H, $Sn(CH_2)_3Me$), 1.18 (m, 18H, HCMe + $CHMe_2$), 1.58 (m, 12H, $Sn(CH_2)_3Me$), 3.78 (br, 4H, CHMe + $CHMe_2$), 4.52 (t, 2H, NCH_2), 5.81 (t, 2H, OCH_2), 7.91-8.08 (m, 4H, aromatic-H), 8.91 (s, 1H, CH=N)	-164	60
14	1614 υ(C=N), 1546 υ(C=C), 964 υ(C-H), 1330 υ($OCMe_2$), 1048 υ(C-O) alcoholic, 1372 υ(C-O) alcoholic(L), 544 υ(Sn-O), 582 υ(Sn-C), 520 υ(Sn-N), 482 υ(Al-O), 1150,1130 υ(OPr^i), 1412 υ(NO_2).	0.91 (m, 6H, $Sn(CH_2)_3Me$), 1.32 (m, 36H, $Sn(CH_2)_3Me$, CMe_2 + $CHMe_2$), 4.50 (m, 4H, NCH_2 + $CHMe_2$), 5.81 (t, 2H, OCH_2), 7.96-8.10 (m, 4H, aromatic-H), 8.98 (s, 1H, CH=N)	-178	55
15	1646 υ(C=N), 1532 υ(C=N) thiol, 1134 υ(C-N), 756 υ(C-S), 1544 υ(C=C), 962 υ(C-H), 1236 υ(C-O) phenolic, 1046 υ(C-O) alcoholic, 542 υ(Sn-O), 584 υ(Sn-C), 522 υ(Sn-N), 482 υ(Al-O), 450 υ(Al-N), 1154,1134 υ(OPr^i), 2950, 2834 υ(CH_2) dea.	0.94 (m, 6H, $Sn(CH_2)_3Me$), 1.23 (m, 24H, $Sn(CH_2)_3Me$ + $CHMe_2$), 3.19 (t, 4H, NCH_2), 3.85 (t, 4H, OCH_2), 4.40 (br, 3H, NH + $CHMe_2$) 7.09-8.18 (m, 8H, aromatic-H), 9.21 (s, 1H, CH=N)	-189	22
16	1642 υ(C=N), 1522 υ(C=N) thiol, 1136 υ(C-N), 755 υ(C-S), 1546 υ(C=C), 964 υ(C-H), 1238 υ(C-O) phenolic, 1047 υ(C-O) alcoholic, 546 υ(Sn-O), 582 υ(Sn-C), 524 υ(Sn-N), 484 υ(Al-O), 454 υ(Al-N), 1151,1132 υ(OPr^i), 2954, 2835 υ(CH_2) dea.	0.89 (m, 6H, $Sn(CH_2)_3Me$), 1.26 (m, 24H, $Sn(CH_2)_3Me$ + $CHMe_2$), 2.32 (S, 3H, NMe) 2.62 (t, 4H, NCH_2), 3.91 (br, 6H, OCH_2 + $CHMe_2$), 7.09-8.16 (m, 8H, aromatic-H), 9.28 (s, 1H, CH=N)	-182	27
17	1618 υ(C=N), 1546 υ(C=C), 960 υ(C-H), 1045 υ(C-O) alcoholic, 1372 υ(C-O) alcoholic(L), 542 υ(Sn-O), 582 υ(Sn-C), 520 υ(Sn-N), 482 υ(Al-O), 452 υ(Al-N), 1151,1130 υ(OPr^i), 1421 υ(NO_2), 2952, 2836 υ(CH_2) dea.	0.93 (m, 6H, $Sn(CH_2)_3Me$), 1.28 (m, 24H, $Sn(CH_2)_3Me$ + $CHMe_2$), 3.28 (m, 8H, NCH_2(L) + NCH_2(dea) + $CHMe_2$), 3.91 (m, 7H, OCH_2 (L) + OCH_2(dea) + NH), 7.10-8.19 (m, 4H, aromatic-H), 9.16 (s, 1H, CH=N)	-178	29
18	1620 υ(C=N), 1546 υ(C=C), 962 υ(C-H), 1046 υ(C-O) alcoholic, 1374 υ(C-O) alcoholic(L), 544 υ(Sn-O), 586 υ(Sn-C), 524 υ(Sn-N), 485 υ(Al-O), 456 υ(Al-N), 1155,1133 υ(OPr^i), 1424 υ(NO_2), 2956, 2832 υ(CH_2) dea.	0.90 (m, 6H, $Sn(CH_2)_3Me$), 1.24 (m, 24H, $Sn(CH_2)_3Me$ + $CHMe_2$), 2.33 (s, 3H, NMe), 3.25 (br, 6H, NCH_2(L) + NCH_2(dea)), 3.93 (m, 8H, OCH_2 (L) + OCH_2(dea) + $CHMe_2$), 7.19-8.23 (m, 4H, aromatic-H), 9.21 (s, 1H, CH=N)	-184	33

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^{119}Sn NMR

^{119}Sn NMR spectroscopy has demonstrated its effectiveness in providing valuable insights into the coordination state of tin atom. The chemical transformation of ^{119}Sn in organotin(IV) derivative is affected by many factors, such as coordination of donor atom, the nature of alkyl group in tin, the properties of the chemical ligands and collection of the derivatives involved. Based on this, the present interpretation is limited to the importance of quality. Five coordination number shown by organotin derivatives in ^{119}Sn NMR lies between -90 to -236 ppm [29-31].

A comparison of ^{119}Sn chemical shifts of (**1**)-(**18**) at δ 165 ± 25 ppm similar to $Bu_2Sn(OCH_2CH_2)_2NR$ (R = H, δ-210; R = Me = δ-205) and diorganotin(IV) n-arylsalicyladiminates (δ -137±6, -170±2), which from a discrete, five coordinated [29-31]. Based on the previous explanation, it can be concluded that all the (**1**)-(**18**) are monomeric type of tin atom with two alkyl groups. In the triangular bipyramidal geometry around it, the nitrogen in the equatorial position and axial position of the two oxygen atom are formed.

^{27}Al NMR

The ^{27}Al NMR signals observed at δ 52, 58, 60 and 55 ppm for (**11**), (**12**), (**13**) and (**14**) on four-coordinated aluminium derivative [33-35]. (**15**), (**16**), (**17**) and (**18**) found signals at δ 22, 27, 29 and 33 ppm, respectively, based on the five coordinate progression [4,13,17,31].

Table 2 Some analytical and physical data for derivatives (**1**) - (**18**)

Reactants (g, mmol)		Product molecular formula (% yield Found, state) (Cal.: g, mmol)	Liberated Pr^iOH (g, mmol)	% Analysis Found (Calc.)					
				Sn	Al	C	H	N	M.wt.Found (Calc.)
$Bu_2Sn(OPr^i)_2$ (8.90, 25.42)	$HOC_6H_4CH=NCNSC_6H_4$ (6.46, 25.42)	{($C_{25}H_{34}N_2O_2S$)Sn }(1) (82, sticky solid) (13.86, 25.42)	(1.52, 25.42)	21.68 (21.77)	-	54.98 (55.06)	6.14 (6.28)	5.01 (5.13)	558 (545)
$Bu_2Sn(OPri)_2$ (8.90, 25.42)	$O_2NC_6H_4CH=NCH_2CH_2OH$ (4.93, 25.42)	{($C_{20}H_{34}N_2O_4$)Sn} (2) (88, sticky solid) (11.92, 25.42)	(1.52, 25.42)	24.35 (24.47)	-	49.45 (49.51)	6.65 (7.06)	5.58 (5.77)	498 (485)
(**1**) (1.88, 3.46)	HOCH(Me)CH(Me)OH (0.31, 3.46)	{($C_{26}H_{36}N_2O_3S$)Sn }(3) (88, sticky solid) (1.99, 3.46)	(0.20, 3.46)	20.55 (20.63)	-	54.12 (54.28)	6.25 (6.31)	4.82 (4.87)	582 (575)
(**1**) (1.88, 3.46)	$HOC(Me)_2C(Me)_2OH$ (0.48, 3.46)	{($C_{28}H_{40}N_2O_3S$)Sn} (4) (85, sticky solid) (2.08, 3.46)	(0.20, 3.46)	19.58 (19.67)	-	55.65 (55.73)	6.62 (6.68)	4.59 (4.64)	612 (603)
(**2**) (2.10, 4.48)	HOCH(Me)CH(Me)OH (0.40, 4.48)	{($C_{21}H_{36}N_2O_5$)Sn} (5) (82, sticky solid) (2.30, 4.48)	(0.26, 4.48)	23.01 (23.04)	-	48.88 (48.95)	6.98 (7.04)	5.35 (5.44)	539 (515)
(**2**) (2.10, 4.48)	$HOC(Me)_2C(Me)_2OH$ (0.53, 4.48)	{($C_{23}H_{40}N_2O_5$)Sn} (6) (80, sticky solid) (2.43, 4.48)	(0.26, 4.48)	21.78 (21.85)	-	50.78 (50.85)	7.35 (7.42)	5.06 (5.15)	562 (543)
(**1**) (1.88, 3.46)	$H\text{-}N(CH_2CH_2OH)_2$ (0.36, 3.46)	{($C_{26}H_{37}N_3O_3S$)Sn.} (7) (84, sticky solid) (2.04, 3.46)	(0.20, 3.46)	20.02 (20.11)	-	52.78 (52.90)	6.22 (6.32)	6.98 (7.11)	598 (590)
(**1**) (1.88, 3.46)	$CH_3\text{-}N(CH_2CH_2OH)_2$ (0.41, 3.46)	{($C_{27}H_{39}N_3O_3S$)Sn} (8) (84, sticky solid) (2.09, 3.46)	(0.20, 3.46)	19.52 (19.64)	-	53.45 (53.65)	6.42 (6.51)	6.82 (6.95)	618 (604)
(**2**) (2.10, 4.48)	$H\text{-}N(CH_2CH_2OH)_2$ (0.47, 4.48)	{($C_{21}H_{37}N_3O_5$)Sn}(9) (85, sticky solid) (2.37, 4.48)	(0.26, 4.48)	22.24 (22.39)	-	47.45 (47.57)	6.92 (7.03)	7.75 (7.92)	552 (530)
(**2**) (2.10, 4.48)	$CH_3\text{-}N(CH_2CH_2OH)_2$ (0.53, 4.48)	{($C_{22}H_{39}N_3O_5$)Sn}(10) (88, sticky solid) (2.43, 4.48)	(0.26, 4.48)	21.72 (21.81)	-	48.42 (48.55)	7..08 (7.22)	7.58 (7.72)	565 (544)
(**3**) (1.99, 3.46)	$Al(OPr^i)_3$ (0.70, 3.46)	{($C_{32}H_{49}N_2O_5S$)Sn.Al}(11) (84, sticky solid) (2.48, 3.46)	(0.20, 3.46)	16.42 (16.50)	3.68 (3.75)	53.35 (53.42)	6.81 (6.86)	3.82 (3.89)	725 (719)
(**4**) (2.08, 3.46)	$Al(OPr^i)_3$ (0.70, 3.46)	{($C_{34}H_{53}N_2O_5S$)Sn.Al}(12) (87, sticky solid) (2.58, 3.46)	(0.20, 3.46)	15.82 (15.88)	3.58 (3.61)	54.55 (54.62)	7.04 (7.15)	3.69 (3.75)	758 (747)
(**5**) (2.30, 4.48)	$Al(OPr^i)_3$ (0.91, 4.48)	{($C_{27}H_{49}N_2O_7$)Sn .Al}(13) (89, sticky solid) (2.95, 4.48)	(0.26, 4.48)	17.95 (18.00)	4.02 (4.09)	49.02 (49.18)	7.38 (7.49)	4.18 (4.25)	672 (659)
(**6**) (2.43, 4.48)	$Al(OPr^i)_3$ (0.91, 4.48)	{($C_{29}H_{53}N_2O_7$)Sn.Al} (14) (83, sticky solid) (3.07, 4.48)	(0.26, 4.48)	17.19 (17.27)	3.85 (3.93)	50.58 (50.67)	7.65 (7.77)	3.98 (4.07)	698 (687)
(**7**) (2.04, 3.46)	$Al(OPr^i)_3$ (0.70, 3.46)	{($C_{32}H_{50}N_3O_5S$)Sn.Al}(15) (86, sticky solid) (2.54, 3.46)	(0.20, 3.46)	16.04 (16.16)	3.52 (3.67)	52.24 (52.33)	6.65 (6.86)	5.58 (5.72)	745 (734)
(**8**) (2.09, 3.46)	$Al(OPr^i)_3$ (0.70, 3.46)	{($C_{33}H_{52}N_3O_5S$)Sn.Al}(16) (87, sticky solid) (2.58, 3.46)	(0.20, 3.46)	15.72 (15.86)	3.48 (3.60)	52.81 (52.95)	6.85 (7.00)	5.48 (5.62)	760 (748)
(**9**) (2.37, 4.48)	$Al(OPr^i)_3$ (0.91, 4.48)	{($C_{27}H_{50}N_3O_7$)Sn .Al}(17) (82, sticky solid) (3.02, 4.48)	(0.26, 4.48)	17.45 (17.61)	3.85 (4.00)	47.95 (48.07)	7.32 (7.48)	6.12 (6.23)	687 (674)
(**10**) (2.43, 4.48)	$Al(OPr^i)_3$ (0.91, 4.48)	{($C_{28}H_{52}N_3O_7$)Sn .Al}(18) (86, sticky solid) (3.08, 4.48)	(0.26, 4.48)	17.08 (17.24)	3.75 (3.92)	48.72 (48.85)	7.54 (7.62)	5.98 (6.10)	697 (688)

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3.2 Biological Activity

The antibacterial properties of Schiff base, diethanolamine, and glycols may be attributed to the presence of hydroxyl groups, nitrogen atoms, and/or azomethine linkages [2,32,33]. It is hypothesized that ethanolamine's antibacterial activity results from either damaging the cell membrane or from its surface-active characteristics. Compared to the parent ligands, all of the complexes exhibited increased antibacterial activity.

3.2.1 Antibacterial Activity

The antibacterial activity of the newly synthesized derivative was tested against *E. coli* [MTCC 433] and *S. aureus* [MTCC 9886] bacterial strains *via* agar-diffusion method and nutrient broth dilution methods. Ciprofloxacin was chosen as standard antibacterial drugs [5,34,35].

3.2.2 Agar Plate Method

The agar suspension was prepared as per the manufacture guidelines and pour into the petri dishes allow it to solidify (**1**). Swab the bacterial culture using sterile loop on the entire surface of the agar plate. Further using sterile metallic borer synthesized derivatives and antibiotics were placed on the nutrient agar plate. All the plates were incubated at in BOD incubators at 37±1°C for 24 h. After incubation, zone of inhibition was determined using a ruler in millimeters (mm).

3.2.3 Tube Dilution Methods

The test derivative's MIC and MBC were ascertained using the tube dilution method [35]. For every experiment, the bacterial strains *S. aureus* and *E. coli* were subcultured for 24 hours at 37 °C.

Autoclaved nutrient broth was used to prepare culture media. Serial of test tube were prepared for each derivative and standards then bacterial suspension were added. All the tubes were incubated at 35-37 °C for 16-20 hours. Bacterial growth inhibition by the test derivatives and ciprofloxacin was analyzed using UV-visible spectroscopy at 600 nm and MIC and MBC was calculated.

Table 3 Zone of inhibition exhibited by tested derivatives and standard

Derivatives	Zone of Inhibition (mm)	
	Agar diffusion method	
	E. coli	*S. aureus*
1. $Bu_2Sn(L^1)(OPr^i)$	11.5 ± 0.19	13.1 ± 0.11
2. $Bu_2Sn(L^2)(OPr^i)$	18.4 ± 0.21	20.3 ± 0.23
3. $Bu_2Sn(L^1)(O\text{-}G^1\text{-}OH)$	19.2 ± 0.15	19.8 ± 0.25
4. $Bu_2Sn(L^1)(O\text{-}G^2\text{-}OH)$	18.9 ± 0.13	19.5 ± 0.23
5. $Bu_2Sn(L^2)(O\text{-}G^1\text{-}OH)$	25.9 ± 0.24	26.7 ± 0.27
6. $Bu_2Sn(L^2)(O\text{-}G^2\text{-}OH)$	25.1 ± 0.18	25.9 ± 0.26
7. $Bu_2Sn(L^1)$(deaH)	17.1 ± 0.12	17.9 ± 0.15
8. $Bu_2Sn(L^1)$(MedeaH)	16.6 ± 0.17	17.3 ± 0.08
9. $Bu_2Sn(L^2)$(deaH)	24.6 ± 0.25	25.4 ± 0.32
10. $Bu_2Sn(L^2)$(MedeaH)	23.1 ± 0.17	23.7 ± 0.27
11. $Bu_2Sn(L^1)\{O\text{-}G^1\text{-}O\text{-}Al(OPr^i)_2\}$	20.8 ± 0.19	21.7 ± 0.16
12. $Bu_2Sn(L^1)\ \{O\text{-}G^2\text{-}O\text{-}Al(OPr^i)_2\}$	19.3 ± 0.14	20.1 ± 0.09
13. $Bu_2Sn(L^2)\ \{O\text{-}G^1\text{-}O\text{-}Al(OPr^i)_2\}$	27.2 ± 0.18	27.9 ± 0.31
14. $Bu_2Sn(L^2)\ \{O\text{-}G^2\text{-}O\text{-}Al(OPr^i)_2\}$	26.2 ± 0.28	26.9 ± 0.23
15. $Bu_2Sn(L^1)\ \{deaAl(OPr^i)_2\}$	18.4 ± 0.18	19.1 ± 0.2
16. $Bu_2Sn(L^1)\{MedeaAl(OPr^i)_2\}$	17.7 ± 0.16	18.2 ± 0.3
17. $Bu_2Sn(L^2)\ \{deaAl(OPr^i)_2\}$	25.4 ± 0.19	26.1 ± 0.21
18. $Bu_2Sn(L^2)\ \{MedeaAl(OPr^i)_2\}$	24.2 ± 0.23	25.1 ± 0.18
19. Ciprofloxacin Standard	26.3± 0.11	22.6 ± 0.09

Table 4 MIC and MBC exhibited by synthesized derivatives

Derivatives	*E. coli*		*S. aureus*	
	MIC (µg/mL)	MBC (µg/mL)	MIC (µg/mL)	MBC (µg/mL)
1. $Bu_2Sn(L^1)(OPr^i)$	35	105	80	200<
2. $Bu_2Sn(L^2)(OPr^i)$	25	65	50	110
12. $Bu_2Sn(L^1)\ \{O\text{-}G^2\text{-}O\text{-}Al(OPr^i)_2\}$	15	45	40	120
14. $Bu_2Sn(L^2)\ \{O\text{-}G^2\text{-}O\text{-}Al(OPr^i)_2\}$	05	30	25	75
16. $Bu_2Sn(L^1)\{MedeaAl(OPr^i)_2\}$	25	75	60	180
18. $Bu_2Sn(L^2)\ \{MedeaAl(OPr^i)_2\}$	15	50	40	90
19. Ciprofloxacin Standard	0.75	1.50	0.25	0.50

All the synthesized derivative were screened for *in vitro* antibacterial activity against *E. coli* [Gram (-)] and *S. aureus* [Gram (+)] at concentration 10 µg/mL. Ciprofloxacin was used as the reference drug. The results of zone of inhibition by synthesized derivatives (**1**)-(**18**) are summarized in Table 3. Interestingly, zone of inhibition by all the selected derivatives is higher for *S. aureus* whereas standard showed higher zone of inhibition towards *E. coli*.

Further in tube dilution assay, IC_{50} was calculated against *E. coli* and *S. aureus* and presented in **Table 4**. The selected derivatives (**1, 2, 12, 14, 16** and **18**) showed potent inhibitory activity against *E. coli*. The bacterial bioassay result was compared with standard drug ciprofloxacin.

4. Conclusion

Heterolaptic homo and heterometallic derivatives were successfully synthesized and characterized with spectroscopic studies. The bipyramidal structure of butyltin is being proposed on above discussed bases. Antibacterial activity of synthesized derivative is compared with Ciprofloxacin taking it as a standard and found that zone of inhibition by all the derivatives is higher zone for *S. aureus* whereas standard showed higher zone of inhibition towards *E. coli*.

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