



## Synthesis, Characterization and Antimicrobial Evaluation of Diorganotin(IV) Complexes of Schiff Base

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### ABSTRACT

A series of diorganotin(IV) complexes of *N*-(2-hydroxy-1-naphthylidene) glycine (**1**), having general formula  $[Me_2SnOPr^i(Hhngl)]$  (**2**),  $[Me_2Sn(Hhngl)_2]$  (**3**),  $[Bu_2SnOPr^i(Hhngl)]$  (**4**),  $[Bu_2Sn(Hhngl)_2]$  (**5**),  $[Ph_2Sn(hngl)]$  (**6**),  $[Ph_2Sn(Hhngl)_2]$  (**7**), [where  $H_2hngl = N$ -(2-hydroxy-1-naphthylidene) glycine] were synthesized by reacting of diorganotin(IV)chloride with the ligand, with the aid of sodium iso-propoxide in appropriate stiochiometric ratios (1:1, 1:2). All the complexes exhibit good antibacterial activity against two Gram-positive bacteria namely, *Staphylococcus aureus*, *Bacillus subtilis* and two Gram-negative bacteria namely, *Escherichia coli* and *Pseudomonas aeruginosa*. All the complexes were also exhibit remarkable antifungal activity against three pathogenic fungi namely, *Aspergillus niger*, *A. flavus* and *Penicillium sp.*

**Keyword:** diorganotin complexes, Schiff base, spectroscopic studies, antifungal activity, antibacterial activity

### 1. INTRODUCTION

The interest in tin chemistry has attracted considerable attention due to its remarkable industrial, medicinal and agricultural applications [1-3] and the coordination mode between tin metal and the ligand has been studied from the last decade due to their versatile and significant biological activities [4-10]. On the other hand, Schiff bases are very important ligands in medicinal and pharmaceutical fields because of their wide spectrum of biological activities. Most of them show biological activities such as antineoplastic, anti-insecticidal [11], antibacterial [12], antifungal [13], anti HIV [14-15], anti-inflammatory [15] as well as antitumor activity [16]. The rapid development of these ligands resulted in an advance research activity in the field of coordination chemistry leading to very interesting conclusions. Prompted by these facts, we have synthesized some diorganotin(IV) complexes, as potential antibacterial and antifungal agents.

### 2. EXPERIMENTAL

#### 2.1. Materials and Methods

All the reagents, viz., 2-hydroxy-1-naphthaldehyde (Aldrich), dimethyltin(IV) dichloride (Merck), dibutyltin(IV) dichloride (Alfa Aesar) and diphenyltin(IV) dichloride (Alfa Aesar) were used as received. All the chemicals and solvents used, were dried and purified by standard methods, and moisture was excluded from the glass apparatus using  $CaCl_2$  drying tubes. The melting points were

determined in open capillaries with electronic melting point apparatus. C, H and N analysis of ligand and complexes were carried on a VarioEL, CHNS elemental analyzer. The tin content in the synthesized complexes were determined gravimetrically as  $SnO_2$ . Infrared spectra of the solid compounds were recorded on a Perkin-Elmer 1600 series FT-IR spectrophotometer in the range 4000-500  $cm^{-1}$  from KBr discs and 500-200 from CsI discs.  $^1H$  NMR spectra were recorded on a Bruker Avance II 400 NMR spectrometer at SAIF, Punjab University, Chandigarh, India, using DMSO or  $CDCl_3$  as a solvent and TMS as the internal standard. The Conductivity Measurement was performed using conductometer Eco Testr EC Low in DMSO having  $10^{-3}$  M at room temp. The antibacterial and antifungal activity of ligand and their diorganotin(IV) complexes were evaluated by the agar well diffusion method and poison food technique, respectively.

#### 2.2. Synthesis of Schiff Base, $H_2hngl$ (**1**)

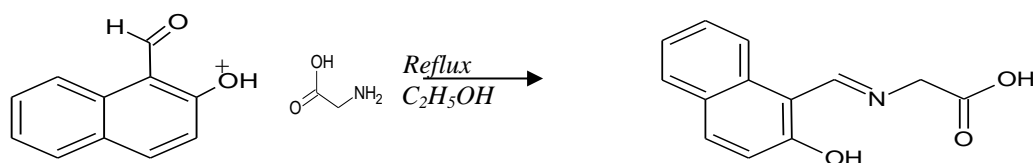
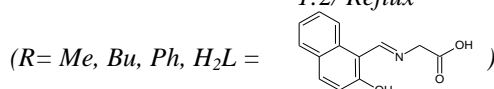
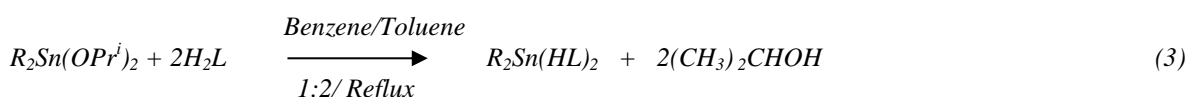
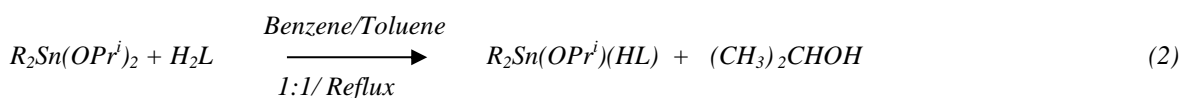
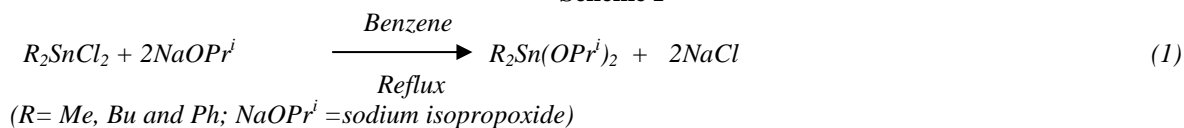
Schiff bases were prepared by condensation of hot aqueous (25 ml) solution of glycine (13 mmol) and 2-hydroxy-1-naphthaldehyde (13 mmol), dissolved in ethanol (50 ml). The reaction mixture was refluxed for about 2 h and yellow brown polycrystalline precipitate was obtained after standing overnight. It was purified by repeated washing with aqueous-ethanol (1: 2) and dried in vacuum over fused  $CaCl_2$  (Scheme 1).

#### 2.3. Synthesis of Complexes

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**Scheme I**

**Scheme II**
**[Me<sub>2</sub>SnOPr<sup>i</sup>(Hhngl)] (2)**

A solution of dimethyltin (IV) dichloride (1.098 g, 5 mmol) in toluene (10 ml) was treated with sodium isopropoxide (0.230 gm, 10 mmole) to produce dimethyltin (IV) diisopropoxide and sodium chloride. The sodium chloride precipitate was removed by filtration and the solvent was removed by distillation. The solution of dimethyltin (IV) diisopropoxide (1.334 g, 5 mmol) and ligand (1.031 g, 4.5 mmol), in toluene, was stirred for half an hour and then refluxed in toluene for 8-12 h. These reactions proceed with the liberation of isopropanol, which is removed azeotropically with benzene under reduced pressure. The oily product thus obtained was solidified and purified by trituration with petroleum ether (b.p. 60-80 °C).

**[Me<sub>2</sub>Sn(Hhngl)<sub>2</sub>] (3)**

Complex **3** was prepared in the similar way as complex **2**. Dimethyltin (IV) diisopropoxide (1.334 g, 5 mmol), ligand (2.063 gm, 9 mmol).

**[Bu<sub>2</sub>SnOPr<sup>i</sup>(Hhngl)] (4)**

Complex **4** was prepared in the similar way as complex **2**. Dibutyltin(IV) diisopropoxide (1.754 g, 5 mmol) ligand (1.031 g, 4.5 mmol), solvent, benzene.

**[Bu<sub>2</sub>Sn(Hhngl)<sub>2</sub>] (5)**

Complex **5** was prepared in the similar way as complex **2**. Dibutyltin(IV) diisopropoxide (1.754 g, 5 mmol), ligand (2.063 gm, 9 mmol), solvent, benzene.

**[Ph<sub>2</sub>SnOPr<sup>i</sup>(Hhngl)] (6)**

Complex **6** was prepared in the similar way as complex **2**. diphenyltin(IV) diisopropoxide (1.955 g, 5 mmol), ligand (1.031 g, 4.5 mmol), solvent, benzene.

**[Ph<sub>2</sub>Sn(Hhngl)<sub>2</sub>] (7)**

Complex **7** was prepared in the similar way as complex **2**. diphenyltin(IV) diisopropoxide (1.955 g, 5 mmol), ligand (2.063 gm, 9 mmol), solvent, benzene.

**3. RESULT AND DISCUSSION**

The Schiff base was prepared by adopting earlier reported method [17]. A new methodology has been used to synthesized diorganotin(IV) complexes. Organotin complexes are usually prepared by reacting organotin hydroxide or dialkyltin oxide to corresponding ligand and also by reacting organotin halide to sodium or potassium salt of ligand. In present study, we have replaced halogen of tin- or organotin with isopropoxide group by reacting them to sodium isopropoxide (Scheme II). The tin- and organotin isopropoxides were isolated and reacted to ligand. These reactions proceed with the liberation of isopropanol, which is fractionated out azeotropically and estimated to monitor the completion of reaction. Owing to highly hydroscopic nature of the alkyl and aryl substituted tin (IV) alkoxides, all the reactions would be carried out under strictly anhydrous condition. The structure of complexes was confirmed by their physio-chemical analysis such as elemental, azeotropic, gravimetric analysis and conductivity measurement. The precise information about their structure is obtained from IR and <sup>1</sup>H NMR spectral measurements.

**3.1. Elemental Analysis**

Experimental and calculated elemental compositions of the complexes are given in Table I. The analytical data are in good agreement with the proposed stoichiometry of the complexes.

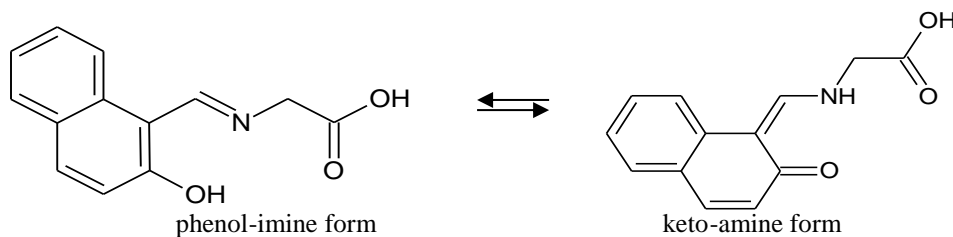


Figure 1. Tautomeric form of Schiff base.

### 3.2. Molar Conductance

Molar conductance of the synthesized complexes showed very low values indicating their non-electrolytic nature [18].

### 3.3. Infrared Spectra

The characteristic infrared frequencies of the diorganotin (IV) complexes are given in table 2. It has been suggested, Schiff bases have a tautomeric structure (Figure 1), which means that Schiff bases exist in keto-amine and phenol-imine forms. But upon complex formation, they may exist only in the imine form [19]. Y. Ozcan et al. have suggested a phenol-imine tautomeric form in the crystal structure of analogous Schiff base [20].

The characteristic infrared frequencies of the diorganotin (IV) complexes are given in table 2. The IR spectra of the diorganotin complexes display a broad vibrational band at 3400 **2**, 3439 **3**, 3400 **4**, 3448 **5**, 3450 **6**, 3420 **7**  $\text{cm}^{-1}$  [21-22], which are assignable to the unbonded -OH stretching of the phenolic group.

These complexes give a strong asymmetric stretching frequencies  $\nu_{as}(\text{COO})$  near 1620-1648  $\text{cm}^{-1}$  and a weaker symmetrical stretching frequencies  $\nu_s(\text{COO})$  near 1390  $\text{cm}^{-1}$ . The magnitude of  $\nu_{as} - \nu_s$  ( $\Delta\nu$ ) separation has been used to explain the type of bonding of carboxylate group to the tin metal [23]. The magnitude of  $\nu_{as} - \nu_s$  ( $\Delta\nu$ ) for these complexes are above 200  $\text{cm}^{-1}$ , indicating the monodentate bonding of the carboxylate group to the tin metal. In diorganotin(IV) complexes,  $\nu(\text{C}=\text{N})$  band, display between 1590-1612  $\text{cm}^{-1}$ , is considerably shifted towards lower frequencies with respect to that of the free Schiff base (around 1620  $\text{cm}^{-1}$ ), indicating the coordination of the azomethine nitrogen to the diorganotin(IV) moiety.

The bonding of the carboxylate group to the tin metal is further confirmed by the appearance of a band at 503-574  $\text{cm}^{-1}$ , assignable to the Sn-O stretching frequency [24]. In the lower frequency region, the band observed in the region 418-470 has been assigned to the  $\nu\text{Sn} \leftarrow \text{N}$  vibration [25-28]. The far IR spectra of  $\text{Ph}_2\text{SnOPr}^i(\text{Hhngl})$  and  $\text{Ph}_2\text{Sn}(\text{Hhngl})_2$  show bands at  $272 \pm 3$  and  $224 \pm 22$   $\text{cm}^{-1}$ , [29] which may be assigned to the  $\nu_{asym}(\text{Sn}-\text{C})$  and  $\nu_{sym}(\text{Sn}-\text{C})$ , respectively, whereas the corresponding peaks at  $593 \pm 17$  and  $522 \pm 3$   $\text{cm}^{-1}$ ,

[29-30] have also been assigned in the spectra of dibutyltin(IV) complexes. In the case of dimethyltin(IV) complexes the appearance of  $\nu$  (Sn-C) bands are not certain due to the overlapping of Sn-O stretching vibration.

### 3.4. $^1\text{H}$ NMR Spectra

Table 3 show the chemical shifts ( $\delta$  in ppm) of various protons in metal complexes. The appearance of a signal at  $\delta$  10.75-10.85 ppm, in the complexes, may be due to the unbonded phenolic -OH proton [31]. The  $^1\text{H}$  NMR Spectra of the complexes, the signals in the region  $\delta$  8.53-9.14 ppm have been assigned to azomethine (-N=CH-) proton [27, 31]. The multiplet between  $\delta$  7.03-8.42 ppm is assigned to the naphthylidene group proton. The butyl protons attached to the tin in dibutyltin(IV) complexes observed at appropriate position in accordance to the previously reported values [27, 32]. In the diphenyltin(IV) complexes, the signals for the phenyl group attached to tin are observed in the range of  $\delta$  =7.06-8.42 ppm, in conjugation with naphthylidene group protons. Two signals due to Sn-Me<sub>3</sub> in dimethyltin(IV) complexes are observed around  $\delta$  =0.83 and  $0.61 \pm 0.03$  ppm [33-34], indicating the presence of methyl group in two different environment. In the light of above finding, the proposed structure of diorganotin(IV) complexes are shown in figure 2.

### 3.5. Biological Activity

Ligand and diorganotin(IV) complexes were screened for their antibacterial and antifungal activity. All these diorganotin(IV) complexes possessed variable antibacterial activity against both Gram-positive (*Staphylococcus aureus* and *Bacillus subtilis*) and Gram negative (*Escherichia coli* and *Pseudomonas aeruginosa*) bacteria and antifungal activity against *Aspergillus niger*, *A. flavus* and *Penicillium* sp. Positive controls produced significantly sized inhibition zones against the tested bacteria and fungi, however, negative control produced no observable inhibitory effect against any of the test organism as shown in Table 4 and 6.

On the basis of maximum inhibitory activity shown against Gram positive bacteria, two complexes (2) and (6) were found to be most effective against *S. aureus* with zone of inhibition of 27.6 mm and 28.6 mm 23.3mm and 24.3mm against *B. subtilis*.

**Table 1.** Elemental analysis of ligand and complexes.

S.N.	Ligand/complexes	Elemental Analysis % Obsd. (Calcd.)				Molar Conductivity ( $\mu\text{S cm}^{-1}$ )
		C	H	N	Sn	
(1)	H <sub>2</sub> L	68.44 (68.12)	4.84 (4.80)	5.89 (6.11)		
(2)	[Me <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	49.58 (49.53)	5.30 (5.27)	3.18 (3.21)	27.26 (27.22)	17
(3)	[Me <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	55.57 (55.52)	4.34 (4.30)	4.64 (4.66)	19.63 (19.61)	10
(4)	[Bu <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	55.40 (55.36)	6.77 (6.73)	2.66 (2.69)	27.85 (27.81)	61
(5)	[Bu <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	59.23 (59.18)	5.56 (5.51)	4.02 (4.06)	17.26 (17.22)	50
(6)	[Ph <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	60.02 (59.98)	3.83 (3.79)	2.76 (2.80)	23.76 (23.73)	42
(7)	[Ph <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	62.58 (62.52)	4.15 (4.11)	3.81 (3.84)	16.30 (16.27)	32

**Table 2.** IR spectra of ligand and complexes (in cm<sup>-1</sup>).

S.N.	Ligand/complexes	$\nu$ (OH)	$\nu$ (C=N)	$\nu_{as}$ (COO)	$\nu_s$ (COO)	$\Delta\nu$ (CO O)	$\nu$ (Sn-O)	$\nu$ (N→S n)	$\nu_{asym}$ (Sn-C)/ $\nu_{sym}$ (Sn-C)
(1)	H <sub>2</sub> L	3500-2300br		1640vs	1397s				
(2)	[Me <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	3400br	1590s	1620vs	1382m	238	571m	442w	533
(3)	[Me <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	3439br	1600s*	1627s	1383shr	244	574br	470w	611
(4)	[Bu <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	3400br	1600s*	1627s	1381m	246	543w	448w	611, 519
(5)	[Bu <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	3448br	1605s*	1627s	1391m	236	503m	418	576, 525
(6)	[Ph <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	3450br	1610vs	1648s	1390m	258	560m	460m	275, 246
(7)	[Ph <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	3420br	1612vs	~1640*	1395m	217	573w	455m	269, 202

**Table 3.** <sup>1</sup>H NMR spectra of ligand and complexes in  $\delta$  (ppm).

S.N.	Ligand/complexes	Ar-OH	-N=CH- proton	naphthylidene group proton	Sn-C <sub>4</sub> H <sub>9</sub> , Sn-CH <sub>3</sub> , Sn-C <sub>6</sub> H <sub>5</sub> protons
(1)	H <sub>2</sub> L		8.60 (s)	7.11-8.34(m)	
(2)	[Me <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	10.83(s)	8.53(s)	7.11-8.38(m)	0.83(s) and 0.64(s)
(3)	[Me <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	10.85(s) and 12.75(s)	8.59(d)	7.03-8.25(m)	0.83(s) and 0.58(s)
(4)	[Bu <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	10.77(s)	9.10(s)	7.06-7.68(m)	1.45-170(m), 1.26(tq) and 0.85(t)
(5)	[Bu <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	10.83(s)	8.90(s)	7.13-8.37(m)	1.75(br), 1.33(m) and 0.85(t)
(6)	[Ph <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	10.76(s)	9.14(s)	7.07-8.30(m)	7.07-8.30(m)
(7)	[Ph <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	10.75(s)	9.10(s)	7.06-8.42(m)	7.06-8.42(m)

s, singlet; t, triplet; q, quartet; br, broad; m, multiplet.

However in case of Gram negative bacteria, again two complexes, (2) and (6) were found to be most active against *E. coli* with the zone of inhibition ranging between 19 mm and 20.6 mm and with zone of inhibition ranging between 16.3mm and 17.3mm against *P. aeruginosa* (Table 4).

In the whole series, the MIC of complexes ranged between 8 and 64  $\mu\text{g/ml}$  against Gram positive bacteria. Complexes (2), (6) and (7) were found to be best as they exhibit the lowest MIC of 8 $\mu\text{g/ml}$

against *S. aureus* and complex (6) against *B. subtilis* with lowest MIC of 16 $\mu\text{g/ml}$ . In case of Gram negative bacteria, MIC of complexes ranged from 64 to 256  $\mu\text{g/ml}$ , complexes (2), (6) and (7) showed lowest MIC of 64 $\mu\text{g/ml}$  against *E. coli* and MIC of 128 $\mu\text{g/ml}$  against *P. aeruginosa* (Table 5). Of the six complexes screened for their antifungal activity, three complexes (2) and (6) showed more than 60% inhibition of mycelial growth against *Aspergillus niger* and complexes (2), (6) and (7) showed more than 60% inhibition of mycelial

growth against *A. flavus* whereas complexes (2), (3) and (6) showed more than 60% inhibition of mycelial growth against *Penicillium* sp. complexes (2) showed highest inhibition of fungal mycelium (68.8%) against *Penicillium* sp. (Table 6).

#### 4. CONCLUSIONS

Based on various studies such as elemental analysis, IR and <sup>1</sup>HNMR spectral studies, five- and six-coordinate geometry for diorganotin(IV) complexes are proposed. The free ligand and their metal complexes were screened against various fungi and bacteria to access their potential as antimicrobial agents. The antimicrobial data reveals that the complexes are superior to the free Ligand. The diorganotin(IV) complexes show remarkable antimicrobial activity. Thus exhibiting their broad spectrum nature and can be further used in pharmaceutical industry for mankind, as an antimicrobial agent, after testing its toxicity to human beings.

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**Table 4:** Antibacterial activity of ligand and complexes through agar well diffusion method.

S. N.	Ligand/complexes	Diameter of growth of inhibition zone (mm) <sup>a</sup>			
		<i>Staphylococcus aureus</i>	<i>Bacillus subtilis</i>	<i>Escherichia coli</i>	<i>Pseudomonas aeruginosa</i>
(1)	H <sub>2</sub> L	17.3 <sup>a</sup> ±0.57 <sup>b</sup>	19.6±1.15	14.6±0.57	12.6±0.57
(2)	[Me <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	27.6±1.15	23.3±0.57	19.0±1	16.3±1.15
(3)	[Me <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	24.3±1.52	22.0±1	16.3±0.57	14.6±1.52
(4)	[Bu <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	22.0±1	21.6±0.57	15.6±0.57	13.3±0.57
(5)	[Bu <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	23.3±0.57	20.6±1.52	15.3±1.15	13.0±1
(6)	[Ph <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	28.6±1.15	24.3±0.57	20.6±1.52	17.3±0.57
(7)	[Ph <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	26.3±0.57	21.6±1.15	18.3±0.57	15.6±1.15
	Ciprofloxacin	27.6±0.57	26.3±1.15	25.0±1	25.0±1

<sup>a</sup>Values, including diameter of the well (8mm), are means of three replicates, <sup>b</sup>± Standard deviation

**Table 5:** Minimum inhibitory concentration (MIC) (in µg/ml) of ligand and complexes by using modified agar well diffusion method.

S. N.	Ligand/complexes	MIC (in µg/ml)			
		<i>Staphylococcus aureus</i>	<i>Bacillus subtilis</i>	<i>Escherichia coli</i>	<i>Pseudomonas aeruginosa</i>
(1)	H <sub>2</sub> L	128	64	256	512
(2)	[Me <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	8	32	64	128
(3)	[Me <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	16	32	128	256
(4)	[Bu <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	32	32	128	256
(5)	[Bu <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	32	64	128	256
(6)	[Ph <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	8	16	64	128
(7)	[Ph <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	8	32	64	128
	Ciprofloxacin	6.5	6.5	6.5	6.5

**Table 6:** Antifungal activity of ligand and complexes through poisoned food method.

S.N.	Ligand/complexes	Mycelial growth inhibition (%)		
		<i>Aspergillus niger</i>	<i>Aspergillus flavus</i>	<i>Penicillium sp.</i>
(1)	H <sub>2</sub> L	51.1±1.1	47.7±1.1	52.5±0.63
(2)	[Me <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	64.7±0.63	66.2±1.68	68.4±1.68
(3)	[Me <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	56.2±1.68	58.4±1.68	61.1±1.1
(4)	[Bu <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	52.9±2.28	50.7±2.28	53.6±0.63
(5)	[Bu <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	55.8±0.63	54.7±0.63	57.7±1.1
(6)	[Ph <sub>2</sub> SnOPr <sup>i</sup> (Hhngl)]	61.1±2.2	64.4±1.1	65.1±1.68
(7)	[Ph <sub>2</sub> Sn(Hhngl) <sub>2</sub> ]	58.4±1.68	62.5±0.63	60.3±0.63
	Fluconazole	81.1±1.1	77.7±1.1	83.3±2.2

± Standard deviation

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