

Synthesis, Characterization, and Biological Activity of Organotin(IV) Complexes with Schiff Bases¹

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Abstract—The synthesized Schiff base ligands H_2L^1 , N,N' -bis(2-hydroxybenzylidene)1,2-diaminobenzene, and H_2L^2 , N,N' -bis(2-hydroxybenzylidene)hydrazine, reacted with Alk_2SnCl_2 to give organotin(IV) complexes **1–10**. The products were characterized by elemental analysis, FTIR, 1H and ^{13}C NMR, and mass spectrometry. All organotin(IV) derivatives with [O,N,O] donor ligands retained their 5-coordinated geometry in solid state and solutions. Most of synthesized complexes exhibited significant antibacterial activity. Cytotoxicity was also tested by using the brine shrimp (*Artemia salina*) lethality bioassay.

Keywords: Schiff base, organotin(IV), IR, NMR, MS, biological activity

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Many organotin compounds of the formula Alk_nSnX_{4-n} demonstrate biological activity in which nature of an alkyl group plays an important role [1, 2]. Activity of those compounds is enhanced by their coordination with Schiff base ligands [3, 4]. Usually tin interaction with Schiff base ligands leads to formation of O–Sn and N–Sn bonds. Such compounds have some potential as antibacterial and antifungal agents [5, 6]. In continuation of our previous study on synthesis of organotin(IV) complexes [7–10], we have synthesized those with Schiff bases to study the coordination behavior of the ligand with the Sn(IV) center. The complexes have been characterized by elemental analysis, IR, 1H , and ^{13}C NMR and mass spectra. Antibacterial activity and cytotoxicity of the complexes was tested.

EXPERIMENTAL

All solvents were obtained from E. Merck Chemicals (Germany) and BDH (England), and purified prior to use according to the standard methods [11]. Most of reagents were purchased from Aldrich chemicals (USA).

Melting points were determined in capillary tubes on a MPD Mitamura Riken Kogyo (Japan) electro-thermal melting point apparatus. FT-IR spectra were recorded (KBr discs) on a Bio-Rad Excaliber FTS Model 3000 MX spectrophotometer. 1H and ^{13}C NMR were measured on a Bruker 300 MHz NMR spectrometer. Elemental analysis was carried out on a CHNS analyzer 932 LECO, USA.

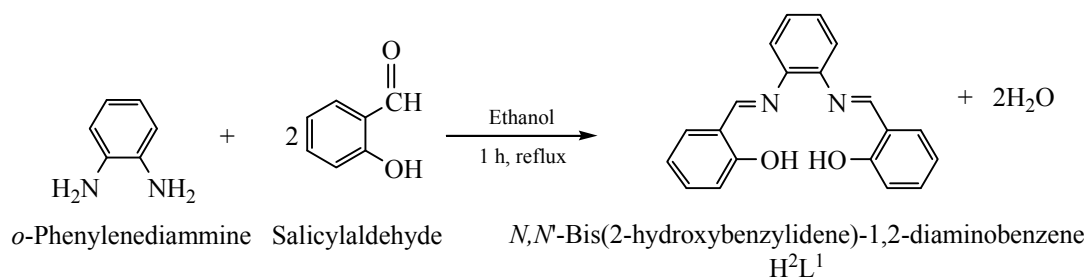
Synthesis of ligands (general procedure). The ligands were synthesized according to the developed earlier methods [12–16] with insignificant modifications.

Synthesis of N,N' -bis(2-hydroxybenzylidene)-1,2-diaminobenzene (H_2L^1). A solution of salicylaldehyde (1 mmol, 12.2 g) in 50 mL of ethanol was slowly added to a solution of *o*-phenylenediamine (5 mmol, 5.4 g) dissolved in 50 mL of ethanol. The reaction mixture was refluxed for 1 h with continuous stirring. The reaction mixture was cooled down, the precipitate was filtered off and washed repeatedly by ethanol and dried in the air (Scheme 1).

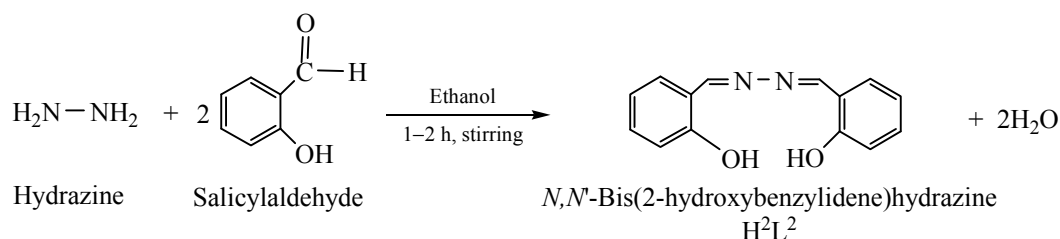
Synthesis of N,N' -bis(2-hydroxybenzylidene)-hydrazine (H_2L^2). A solution of hydrazine (5 mmol) in 50 mL of ethanol was slowly added to a solution of salicylaldehyde (1 mmol) in 50 mL of ethanol. Stirring

¹ The text was submitted by the authors in English.

Scheme 1.



Scheme 2.

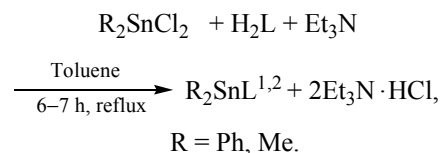


of the reaction mixture lasted for 1–2 h. The corresponding light yellow precipitate was filtered off and washed repeatedly by distilled water, then with ethanol, and dried in the air (Scheme 2).

Synthesis of organotin(IV) complexes 1–10 (general procedure). Diorganotin(IV) complexes **1–10** were prepared by treating the corresponding the ligands with organotin oxides/hydroxides or chlorides [17].

Procedure 1 [based on organotin(IV) chlorides]. A mixture of a Schiff base $\text{H}_2\text{L}^{1,2}$ with R_2SnCl_2 (R = Me, Ph) and Et_3N (ratio 1 : 1 : 2) was refluxed in dry toluene for 6–7 h. After cooling to room temperature, Et_3NHCl formed was filtered off and toluene was

evaporated under reduced pressure. The product obtained was washed, dried and recrystallized from chloroform : *n*-hexane mixture (4 : 1).



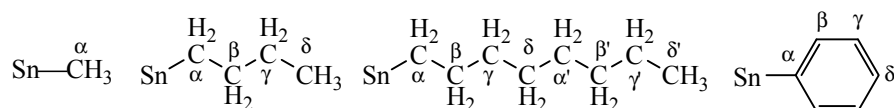
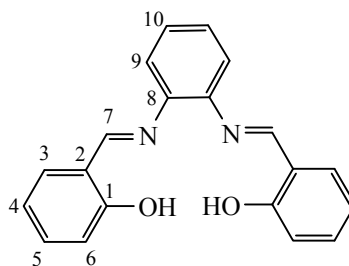
Procedure 2 [based on organotin(IV) oxide/hydroxide]. A mixture of a Schiff base $\text{H}_2\text{L}^{1,2}$ with R_2SnO (R = Bu, Oct) was refluxed (ratio 1 : 1) in dry toluene using a Dean-Stark apparatus for 6–7 h with

Table 1. Physical properties of ligands and their organotin(IV) derivatives

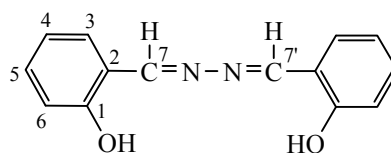
Comp. no	Formula	M_w	mp, °C	Found, %			Calculated, %		
				C	H	N	C	H	N
H_2L^1	$\text{C}_{20}\text{H}_{16}\text{N}_2\text{O}_2$	316	140–142	75.90	5.02	8.82	75.94	5.06	8.86
H_2L^2	$\text{C}_{14}\text{H}_{12}\text{N}_2\text{O}_2$	240	215–216	70.04	5.04	11.62	70.00	5.00	11.66
1	$\text{C}_{22}\text{H}_{20}\text{N}_2\text{O}_2\text{Sn}$	463	180–181	57.05	4.35	6.00	57.01	4.31	6.04
2	$\text{C}_{28}\text{H}_{32}\text{N}_2\text{O}_2\text{Sn}$	547	165–166	61.46	5.89	5.15	61.42	5.85	5.11
3	$\text{C}_{36}\text{H}_{48}\text{N}_2\text{O}_2\text{Sn}$	659	110–112	65.51	7.24	4.20	65.55	7.28	4.24
4	$\text{C}_{32}\text{H}_{24}\text{N}_2\text{O}_2\text{Sn}$	587	80–82	65.45	4.04	4.81	65.41	4.08	4.77
5	$\text{C}_{24}\text{H}_{23}\text{N}_2\text{O}_2\text{ClSn}$	525	240–241	54.81	4.34	5.37	54.85	4.38	5.33

Table 2. IR characteristic bands (cm⁻¹) for ligands and their organotin(IV) derivatives

Comp. no.	$\nu(\text{C}=\text{N})$	$\nu(\text{C}-\text{O})$	$\nu(\text{C}=\text{C})$	$\nu(\text{O}-\text{H})$ H-bonded	$\nu(\text{Sn}-\text{O})$	$\nu(\text{Sn}-\text{N})$	Comp. no.	$\nu(\text{C}=\text{N})$	$\nu(\text{C}-\text{O})$	$\nu(\text{C}=\text{C})$	$\nu(\text{O}-\text{H})$ H-bonded	$\nu(\text{Sn}-\text{O})$	$\nu(\text{Sn}-\text{N})$
H_2L^1	1618	1277	1561	3419	—	—	5	1607	1258	1531	—	538	447
H_2L^2	1624	1267	1574	3200	—	—	6	1610	1240	1580	—	565	461
1	1605	1250	1578	—	540	450	7	1614	1244	1573	—	558	456
2	1600	1256	1525	—	531	484	8	1609	1250	1572	—	562	439
3	1609	1245	1532	—	551	463	9	1612	1253	1575	—	548	450
4	1610	1253	1576	—	546	480	10	1615	1248	1574	—	555	445

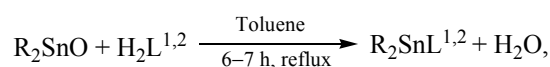
Table 3. ¹H NMR data for *N,N*-bis(2-hydroxybenzylidene)-1,2-diaminobenzene and its organotin(IV) derivatives

Proton	H_2L^1	1	2	3	4	5
OH	13.1 s	—	—	—	—	—
2	7.39 d (3.9)	7.45 d (3.6)	7.33 d (3.5)	7.41 d (3.2)	7.51 d (3.5)	7.26 d (3.8)
3	6.84 t	6.64 t	6.59 t	6.59 t	6.50 t	6.64 t
4	7.23–7.27 m	7.41 t	7.36 t	7.36 t	7.49 t	7.9 t
5	7.07 d (6.9)	7.05 d (7.0)	6.70 d (6.7)	6.77 d (6.5)	6.75 d (6.8)	6.98 d (6.4)
6	8.64 s	8.88 s	8.73 s	8.83 s	8.77 s	8.89 s
7	7.34 d (3.6)	7.55 d (3.5)	7.33 d (3.2)	7.37 d (3.3)	7.39 d (3.4)	7.24 d (3.1)
8	7.23–7.27 m	7.25–7.35 m	7.37–7.39 m	7.20–7.23 m	7.42–7.46 m	6.63–6.69 m
9		0.74 s	1.37–1.43 m	1.36–1.40 m		1.77 t
10			1.27 t	1.21–1.29 m	7.39 d	1.30–1.32 m
α			1.08–1.15 m	1.17–1.24 m	7.25 t	0.79–0.84 m
β			0.64 t	1.17–1.24 m	7.02–7.13 m	0.56–0.60 m
γ				1.17–1.24 m		
δ				1.17–1.24 m		
α'				1.04–1.06 m		
β'				0.83 t		
γ'				26.5		
δ'				22.7		

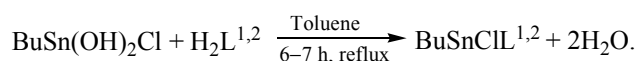
Table 4. ^1H NMR data for *N,N*-bis(2-hydroxybenzylidene)hydrazine and its organotin(IV) derivatives

Proton	H_2L^2	6	7	8	9	10
OH	11.41 s	—	—	—	—	—
3	7.39–7.41 m	7.35–7.44 m	7.36–7.44 m	7.36–7.44 m	7.47–7.5 m	7.36–7.44 m
4	6.99 t (7.2)	6.96 t	6.99 t	6.99 t	6.99 t	6.98 t
5	7.44–7.48 m	7.35–7.44 m	7.36–7.44 m	7.36–7.44 m	7.68–7.7 m	7.36–7.44 m
6	7.05 d (6.5)	7.05 d (6.8)	7.05 d (6.2)	7.05 d (6.9)	7.02 d (6.7)	7.05 d (6.3)
7	8.73 s	8.91 s, 8.74 s	8.89 s, 8.70 s	8.93 s, 8.73 s	8.88 s, 8.71 s	8.79 s, 8.72 s
9		0.8 s	1.38–1.36 m	1.30–1.62 m	—	1.82 t
10			1.13 t	1.30–1.62 m	7.39 d	1.37–1.49 m
α			0.96–0.92 m	1.30–1.62 m	7.42 t	0.90–1.00 m
β			0.96–0.92 m	1.30–1.62 m	7.47–7.5 m	1.0–0.90 m
γ				1.30–1.62 m		
δ				1.30–1.62 m		
α'				1.30–1.62 m		
β'				1.30–1.62 m		
γ'				1.04–1.06 m		
δ'				0.83 t		

continuous removal of water formed. After cooling to room temperature, the solvent was evaporated under reduced pressure. The solid product obtained was air dried and recrystallized from dry chloroform and *n*-hexane mixture (4 : 1).



R = Bu, Oct.



RESULTS AND DISCUSSION

Physical data for the ligands H_2L^1 , H_2L^2 and the synthesized complexes are presented in Table 1. The ligands and the complexes were stable compounds soluble in common organic solvents.

IR spectra. The characteristic IR absorption bands for the ligands and their organotin(IV) complexes are presented in Table 2.

Complexation of tin atom with the ligands was confirmed by the absence of the broad bands, $\nu(\text{OH})$, at 3419 and 3200 cm^{-1} [18]. Stretching vibrations of (C=N) shifted to lower values indicating coordination of tin to nitrogen of a Schiff base in all complexes [19]. Appearance of new bands in the range 565–531 and 484–450 cm^{-1} indicated the presence of Sn–O and Sn–N stretching vibrations, respectively, confirming coordination of tin with N and O atoms [20]. Both ligands bands at 1277 and 1267 cm^{-1} (C–O) were shifted towards lower values in the spectra of complexes due to attachment of tin to oxygen [21]. The shift of C=N band of complexes to lower frequencies indicated donation of nitrogen lone pair of electrons of the azomethine group towards Sn atom [22].

^1H NMR spectra. The signals of aromatic protons of ligands did not show any significant shift for tin

Table 5. ^{13}C NMR data for ligand H_2L^1 and complexes

Carbon	H_2L^1	1	2	3	4	5
1	163.7	175.6	173.5	170.5	169.5	174.4
2	119.0	118.3	117.9	117.9	117.7	119.1
3	132.3	130.1	136.1	136.1	136.1	136.3
4	119.2	121.3	119.7	119.7	119.7	120.2
5	133.4	132.4	137.2	137.2	137.1	137.8
6	117.5	115.6	114.9	114.9	115.6	117.8
7	161.4	166.9	168.6	165.6	163.3	167.7
8	142.5	140.2	140.8	140.9	139.3	138.1
9	119.7	123.8	124.3	124.3	124.6	123.3
10	127.8	128.8	128.3	128.3	128.5	129.9
α		28.4	26.3	25.8	129.1	27.8
β			26.1	22.6	135.2	27.3
γ			18.1	33.2	128.9	26.0
δ			13.6	28.9	127.8	13.9
α'				29.0		
β'				31.8		
γ'				26.5		
δ'				22.7		

Table 6. ^{13}C NMR data for ligand H_2L^2 and complexes

Carbon	H_2L^2	6	7	8	9	10
1	164.6	175.2	179.7	178.7	176.7	177.7
2	117.4	118.6	117.3	117.3	117.3	117.3
3	132.6	131.3	132.6	132.6	132.6	132.6
4	119.3	120.0	119.8	119.8	119.8	119.8
5	133.3	133.7	133.5	133.5	133.5	133.5
6	117.2	116.9	117.2	117.2	117.2	117.2
7, 7'	160.4	168.1, 159.1	166.0, 159.8	170.1, 159.8	169.3, 160.8	168.9, 159.8
α		28.5	28.2	25.7	130.5	28.2
β			27.8	22.7	136.2	26.4
γ			29.7	34.4	129.6	29.7
δ			13.9	29.8	129.2	13.6
α'				32.0		
β'				32.2		
γ'				29.4		
δ'				22.8		

Table 7. Mass spectra data for H₂L¹ and its Sn(IV) derivatives

Fragment ion	1	2	3	4	5
	<i>m/z</i> (%)				
[C ₂₀ H ₁₄ N ₂ O ₂ SnR] ⁺	210 (100)	491 (100)	547 (100)	511(100)	468.9 (100)
[C ₂₀ H ₁₄ N ₂ O ₂ Sn] ⁺	434 (3.5)	434 (17.14)	434 (22.29)	434 (6.15)	434 (33.87)
[C ₁₃ H ₉ N ₂ OSn] ⁺	329 (3.5)	329 (13.56)	329 (27.17)	329 (12.54)	329 (23.58)
[C ₁₃ H ₈ NOSn] ⁺	314 (8.29)	314 (12.06)	314 (9.79)	314 (10.12)	314 (13.62)
[C ₇ H ₄ NOSn] ⁺	238 (6.18)	238 (21.65)	238 (16.97)	238 (16.85)	238 (33.57)
[N ₂ O ₂ Sn] ⁺	180 (12.22)	180 (7.95)	180 (6.35)	180 (11.35)	180 (9.65)
[C ₁₃ H ₁₀ NO] ⁺	196 (32.64)	196 (3.92)	196 (2.0)	196 (8.83)	196 (5.92)
C ₆ H ₅ ⁺	77 (62.0)	77 (7.38)	77 (9.17)	77 (64.13)	77 (32.85)
[Sn] ⁺	120 (21.87)	120 (7.13)	120 (6.5)	120 (20.63)	120 (17.92)

Table 8. Mass spectra data for H₂L² and its Sn(IV) derivatives

Fragment ion	6	7	8	9	10
	<i>m/z</i> (%)				
[C ₁₄ H ₁₀ N ₂ O ₂ Sn] ⁺	358 (35.5)	358 (41.5)	358 (25.2)	358 (39.6)	358 (52.6)
[C ₇ H ₇ N ₂ OSn] ⁺	255 (17.02)	255 (2.98)	255 (15.98)	255 (22.96)	255 (6.43)
[C ₇ H ₅ NOSn] ⁺	239 (13.09)	239 (45.11)	239 (14.65)	239 (12.47)	239 (12.72)
[C ₇ H ₅ OSn] ⁺	225 (9.32)	225 (3.99)	225 (11.58)	225 (7.24)	225 (2.71)
[C ₆ H ₅ OSn] ⁺	213 (2.73)	213 (8.54)	213 (4.17)	213 (3.91)	213 (2.27)
[C ₁₄ H ₁₂ N ₂ O ₂] ⁺	240 (100)	240 (100)	240 (100)	240 (100)	240 (100)
[C ₁₄ H ₁₁ N ₂ O] ⁺	223 (50.20)	223 (5.63)	223 (48.23)	223 (50.98)	223 (46.65)
[[C ₈ H ₇ N ₂ O] ⁺	147 (39.96)	147 (3.22)	147 (37.20)	147 (41.01)	147 (31.59)
[C ₆ H ₅ O] ⁺	93 (54.18)	93 (7.32)	93 (62.20)	93 (71.24)	93 (37.60)
C ₆ H ₅ ⁺	77 (23.12)	77 (8.36)	77 (26.34)	77 (41.07)	77 (17.69)
[Sn] ⁺	120 (50.85)	120 (6.29)	120 (50.15)	120 (59.76)	120 (36.09)

Table 9. *In vitro* antibacterial bioassay of H₂L¹ and its Sn(IV) derivatives

Bacteria	Zone of inhibition, mm						Zone of inhibition of reference drug, mm
	H ₂ L ¹	1	2	3	4	5	
<i>Escherichia coli</i>	–	–	16	–	16	–	25
<i>Bacillus subtilis</i>	–	–	20	20	20	–	26
<i>Shigella flexenari</i>	–	–	13	–	15	–	24
<i>Staphylococcus aureus</i>	–	–	17	–	–	–	17
<i>Pseudomonas aeruginosa</i>	–	–	16	15	16	–	17
<i>Salmonella typhi</i>	–	–	22	20	22	14	21

Table 10. *In vitro* antibacterial bioassay of H_2L^2 and its Sn(IV) derivatives

Bacteria	Zone of inhibition, mm						Zone of inhibition of reference drug, mm
	H_2L^2	6	7	8	9	10	
<i>Escherichia coli</i>	–	–	–	–	13	–	25
<i>Bacillus subtilis</i>	–	–	18	–	20	–	26
<i>Shigella flexenari</i>	–	–	–	–	–	–	24
<i>Staphylococcus aureus</i>	–	–	–	–	–	–	17
<i>Pseudomonas aeruginosa</i>	–	–	12	–	–	–	17
<i>Salmonella typhi</i>	–	–	13	–	17	14	21

Table 11. Brine shrimp (*Artemia salina*) lethality bioassay of H_2L^1 and its Sn(IV) derivatives

Comp. no	Dose, mg/mL	No of shrimps	No of survivors	LD ₅₀ , mg/mL	Standard drug	LD ₅₀ , µg/mL
H_2L^1	100	30	12	32.50	Etoposide	7.46
	10	30	18			
	1	30	24			
1	100	30	13	38.64	Etoposide	7.46
	10	30	18			
	1	30	21			
2	100	30	21	–	Etoposide	7.46
	10	30	24			
	1	30	27			
3	100	30	4	6.02	Etoposide	7.46
	10	30	15			
	1	30	21			
4	100	30	21	–	Etoposide	7.46
	10	30	23			
	1	30	27			
5	100	30	21	–	Etoposide	7.46
	10	30	24			
	1	30	27			

complexes (Tables 3, 4) [23, 24]. In the spectra of both ligands (H_2L^1 , H_2L^2), the hydroxyl group was recorded by signals at 13.1 ppm and 11.41 ppm, respectively, that were absent in the spectra of complexes suggesting the replacement of the phenolic proton [25]. In H_2L^1 , the methine proton (CH=N) signal at 8.64 ppm was recorded in the range of 8.70–8.93 ppm in the spectra of complexes, which confirmed coordination of nitrogen with Sn(IV) [26].

For H_2L^2 , the methine proton signal was recorded at 8.73 ppm as a singlet whereas in the spectra of complexes **6–10** there were recorded two signals for methine proton, which prompted that one of two adjacent nitrogen atoms was involved in the coordination.

^{13}C NMR spectra. The ^{13}C NMR spectral data (Tables 5, 6) for R groups (Me, *n*-Bu, *n*-Oct, and Ph) attached to Sn atom were interpreted by comparison

Table 12. Brine shrimp (*Artemia salina*) lethality bioassay for H₂L² and its Sn(IV) derivatives

Comp. no	Dose, mg/mL	No of shrimps	No of survivors	LD ₅₀ , mg/mL	Standard drug	LD ₅₀ , µg/mL
H ₂ L ²	100	30	21	–	Etoposide	7.46
	10	30	23			
	1	30	27			
6	100	30	16	–	Etoposide	7.46
	10	30	21			
	1	30	24			
7	100	30	18	–	Etoposide	7.46
	10	30	21			
	1	30	25			
8	100	30	16	–	Etoposide	7.46
	10	30	24			
	1	30	27			
9	100	30	1	0.0052	Etoposide	7.46
	10	30	3			
	1	30	5			
10	100	30	8	33.5200	Etoposide	7.46
	10	30	24			
	1	30	27			

with the analogues as model compounds [27]. In H₂L¹ spectra the C¹ appeared at 163.7 ppm whereas in the spectra of complexes it was recorded with a downfield shift induced by deshielding influence of tin which confirmed bonding of oxygen with tin [28]. The signal of C⁷ demonstrated the maximum downfield shift due to participation of nitrogen in coordination with tin. The remaining carbons signals of the complexes appeared almost in the same positions as in the ligand spectra.

The C¹ signal of H₂L² recorded at 164.6 ppm shifted downfield in the spectrum of complexes due to the deshielding effect [29]. Azomethine C^{7,7'} signal of the ligand at 160.4 ppm shifted upon complexation. Two signals were observed for both azomethine carbons because one of two nitrogen atoms was coordinated with tin metal, so the carbon associated with coordinating nitrogen gave the signal downfield due to desheilding.

Mass spectrometry. Mass spectra of all synthesized complexes were recorded using the electron ionization (EI) and the main ion fragments data are presented in Tables 7, 8. Molecular ion peaks were not observed for

complexes **1–10**. The base peaks for complexes **1–5**, and **6–10** were due to the fragments [C₂₀H₁₄N₂O₂SnR]⁺ and [C₁₄H₁₂N₂O₂]⁺, respectively. The peak at 120 assigned to [Sn]⁺⁺ was also observed [29].

Antibacterial activity. The synthesized complexes were screened for antibacterial activity by the agar well diffusion method [30]. The zones of inhibition diameter were measured in mm. The reference drug was Imipenem (Tables 9, 10).

Most of synthesized compounds demonstrated antibacterial activity against the tested bacterial strains more significant than the free ligands [29].

Cytotoxicity. Cytotoxicity of the synthesized compounds was studied by the brine-shrimp lethality method [30]. Brine-shrimp (*Artemia salina*) eggs were hatched in artificial sea water (3.8 sea salt/L) at room temperature (22–29°C). After two days the shrimps were transferred to vials containing 5 mL of artificial sea water (30 shrimp per vial) with final concentrations of each compound 10, 100, 1000 ppm in DMSO. After 24 h the number of survived shrimps was counted.

Data was analyzed with a finny computer programmer to determine LD₅₀ values [4] (Tables 11, 12).

CONCLUSIONS

Complexation of tin with the Schiff base ligands is confirmed by the absence of the broad band characteristic for $\nu(\text{OH})$, and the shift of $(\text{C}=\text{N})$ band to lower value in IR spectra. All organotin(IV) derivatives containing $[\text{O}, \text{N}, \text{O}]$ donor ligands exhibited 5-coordinated geometry in solid state and in solutions. Most of complexes exhibited significant antibacterial activity. Organotin(IV) complexes demonstrated activity against tested brine shrimp larvae.

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