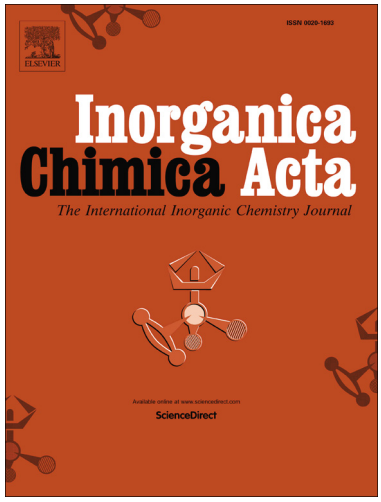

Research paper

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Mehwish Mehmood, Imtiaz-ud-Din, Muhammad Nawaz Tahir, Ihsan-ul Haq, Syeda Saniya Zahra

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Synthetic Stratagem, Characterization and Biocidal applications of Triorganotin(IV) Complexes derived from Hydrazide/Hydrazone Analogues

Mehwish Mehmood[a], Imtiaz-ud-Din[a*], Muhammad Nawaz Tahir[b], Ihsan-ul Haq[c], Syeda Saniya Zahra[c]

Corresponding Author: drimtiazuddin@yahoo.com

[a] Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan.
[b] Department of Physics, University of Sargodha, Sargodha 40100, Pakistan.
[c] Department of Pharmacy, Quaid-i-Azam University, Islamabad 45320, Pakistan.

Abstract

A series of Schiff base ligands as benzylidene benzohydrazide and their hydrazone analogues **(I_a-I_c)** have been synthesized and then complexed with organotin(IV) moiety having general formula [R'_3SnL] to get the target compounds**(1-9)**, where L= $C_4H_3OCONHN{=}CHR$, R= C_6H_5O(**I_a**), C_4H_5O(**I_b**), $C_6H_3Cl_2$(**I_c**) and R'= -CH_3(**1-3**), -CH_2Ph(**4-6**), -Ph(**7-9**). They were fully characterized using FT-IR, NMR(^1H, ^{13}C and ^{119}Sn) spectroscopy, along with elemental analysis and melting point. One of the precursor **(I_b)** has been analyzed by single crystal XRD to further authenticate the structure. The ^{119}Sn NMR data suggest the molecular geometry of organotin derivatives as distorted pentagonal bipyramidal. They were also evaluated for their antibacterial, antifungal, α-amylase inhibition, DPPH, total reducing power and total antioxidant activities. Triaryltin(IV) derivatives show good antibacterial and antifungal activity. Whereas the data for antidiabetic activity demonstrated that the compounds may serve as moderately effective alpha amylase inhibitor.

Keywords: Organotin(IV), Hydrazone, Spectroscopy, X-ray, Alpha Amylase inhibition.

1) Introduction

Organotin(IV) complexes, derived from Schiff bases, have got tremendous importance because of their diverse applications in medicinal and non-medicinal fields[1, 2]. Hydrazides and hydrazones functionalities are considered to be associated with antibacterial[3], antifungal[4], antiplatelet[5] ,anticancer and anti-inflammatory activities[6]. A large number of Schiff bases possess some interesting properties and have been applied in various fields such as their catalytic role in a variety of reactions like hydrogenation, their ability to bind reversibly with oxygen[7] ,as well as studies related to fluorescent, photochromic properties[8] and specifically due to their complexing and chelating ability towards some metals[9].

The present invoke in organotin chemistry comes up with their insecticidal [10], anti-inflammatory[11] and antimicrobial [12] activity. In perpetuation to our previous work [13, 14], we report here the synthesis, spectral studies and variety of biological applications of benzylidene benzohydrazide ligands **(I_a-I_c)** and their organotin derivatives **(1-9)**. The bioactivity of organotin complexes may be affected by the nature and number of donor species, structure as well as their coordination number[15-22]. The effectiveness was further dictated by the presence of specific substituents on the triorganotin moiety. The organotin moiety effects the inhibition of bacterial species due to the attachment of triaryltin moiety, which increases the activity of complex exhibiting MIC values extremely low in mM as compare to the ligands[15]. The data suggest that certain compounds could provide a basic scaffold for optimized antibacterial agents and need to be bioassayed further for discovering potential therapeutic agent in future drug discovery process[23].

2) Experimental Section

2.1 Materials and methods

2-Furoic acid hydrazide, 3-hydroxybenzaldehyde, furaldehyde, 2,4-dichlorobenzaldehyde, trimethyltin chloride and triphenyltin chloride were commercially available and were used as received. Tribenzyltin chloride was prepared as reported earlier[24]. All the solvents used dried using known procedure[25]. The melting points were determined by electro thermal melting point apparatus (model MP-D Mitamura Riken Kogyo Japan) and were uncorrected. The IR spectra were recorded with Bio-Rad Excalibur (model FTS 3000 MX), on KBr discs. The multinuclear (^{1}H, ^{13}C and ^{119}Sn) NMR spectra were recorded on Bruker Advance Digital 300 MHz FT-NMR spectrometer (Switzerland) in deuterated methanol and DMSO as solvents relative to Me_4Si and Me_4Sn as internal reference. The X-ray structure were determined by using Olex2 [26], the structure was refined with ShelXL-1997 refinement package and solved with the ShelXT structure solution program.

2.2 Synthesis of Precursor

The precursors **(Ia-Ic)** as benzylidene benzohydrazide were prepared by reacting stoichiometric amounts of 2-furoic acid hydrazide and the respective substituted aromatic aldehydes, which were dissolved in freshly dried ethanol. The reaction mixture was refluxed for 3-4 hours with constant stirring. The resultant solution was cooled to room temperature with subsequent rotary

evaporation to get the crude product. Recrystallization was carried out in dichloromethane and petroleum ether mixture(1:3) to yield the purified product [27].

2.2.1 (3-hydroxybenzylidene)furan-2-carbohydrazide (I_a)

Quantities used; 2-furoic acid hydrazide(0.25g,2mmole) and 3-hydroxybenzaldehyde(0.24g, 2mmole). White crystals, Yield: 85% ; Melting point: 208-210 ^{0}C; Anal. Calcd. for $C_{10}H_{10}N_2O_3$ (230.22): C, 62.60; H, 4.38; N, 12.17%. Found: C, 62.56; H, 4.31; N, 12.19%; FT-IR Data (KBr, cm^{-1}) $\nu_{(OH)}$ 3265, $\nu_{(NH)}$ 3119, $\nu_{(C=O)}$ 1648, $\nu_{(C=N)}$ 1589, $\nu_{(N-N)}$ 1012, $\nu_{(C-O-C)}$ 1288.; ^{1}H NMR Data (DMSO, δ, ppm) 11.81(1H, s, OH), 9.65(1H, s, NH), 8.36(1H, s, N=CH), 7.95-6.70(7H, m, Ar-H); ^{13}C NMR Data (DMSO, δ, ppm) 158.1 (CO=N), 154.6(N=CH), 147.0, 146.3, 113.0, 112.5(furan-C), 148.3, 148.4,135.9, 10.3, 117.9,115.3 (Ar-C).

2.2.2 (furan-3-ylmethylene)furan-2-carbohydrazide (I_b)

Quantities used; 2-furoic acid hydrazide(0.25g, 2mmoles), furaldehyde(0.16mL,2mmole), brown crystals, Yield: 90% ; Melting point: 192-195 ^{0}C; Anal. Calcd. for $C_{10}H_8N_2O_3$ (204.18): C, 58.82; H, 3.95; N, 13.72%. Found C, 58.84; H, 3.89; N, 13.74%; FT-IR Data (KBr, cm^{-1}) $\nu_{(NH)}$ 3268, $\nu_{(C=O)}$ 1649, $\nu_{(C=N)}$ 1575, $\nu_{(N-N)}$ 1027, $\nu_{(C-O-C)}$ 1291; ^{1}H NMR Data (DMSO, δ, ppm) 11.83(1H, s, NH), 8.33(1H, s, N=CH), 7.94-6.63(6H, m, Ar-H); ^{13}C NMR Data (DMSO, δ, ppm) 156.8 (CO=N), 149.8(N=CH), 147.0, 146.3, 113.0, 112.5(furan-C), 148.3, 148.4,135.9, 10.3, 117.9, 115.3 (Ar-H).156.8 (CO=N), 149.8 (N=CH), 147.05, 146.28, 145.69, 138.03, 115.48, 114.06, 112.69, 112.60 (furan-C).

2.2.3 (2,4-dichlorobenzylidene)furan-2-carbohydrazide (I_c)

Quantities used; 2-furoic acid hydrazide(0.25g, 2mmoles), 2,4-dichlorobenzaldehyde(0.35g, 2mmole), White product; Yield: 80% ; Anal. Calcd. for $C_{12}H_8Cl_2N_2O_2$ (282): C, 50.91; H, 2.85; N, 9.89%. Found C, 50.87; H, 2.92; N, 9.86%; Melting point: 135-137 ^{0}C; FT-IR Data (KBr, cm^{-1}) $\nu_{(NH)}$ 3267, $\nu_{(C=O)}$ 1647, $\nu_{(C=N)}$ 1577, $\nu_{(N-N)}$ 1014, $\nu_{(C-O-C)}$ 1292 ; ^{1}H NMR Data (DMSO, δ, ppm) 11.28(1H, s, NH), 8.87(1H, s, N=CH), 8.15-6.67(6H, m, Ar-H); ^{13}C NMR Data (DMSO, δ, ppm) 156.8 (CO=N), 149.8(N=CH), 145.5, 142.3,115.3, 112.0 (furan-C), 135.6, 134.3, 131.0, 129.3, 128.2, 127.8 (Ar-C).

2.3 Synthesis of targeted compounds (1–9)

Stoichiometric amount of the respective ligands (1mmole) was dissolved in 10mL methanolic solution of potassium hydroxide (1mmole) and refluxed for an hour to get potassium salt of

hydrazone ligand. Then added the appropriate organotin(IV) chlorides into the above solution. The mixture was refluxed with constant stirring for 8-10 hours. The clear solution thus obtained was rotary evaporated to get the targeted compound. They were recrystallized in suitable solvent[15].

2.3.1 [(3-hydroxybenzylidene)furan-carbohydrazide] Trimethyltin(IV) (1)

Quantities used; (3-hydroxybenzylidene)furan-2-carbohydrazide (0.204g, 1mmole), potassium hydroxide (0.1g,1mmole), $(CH_3)_3SnCl$ (0.2g, 1mmole). Dark brown product; Yield: 70% ; Melting point: 182-185 ^{0}C; Anal. Calcd. for $C_{15}H_{18}N_2O_3Sn$(393): C, 45.84; H, 4.62; N, 7.13%. Found C, 45.87; H, 4.53; N, 7.11%; FT-IR Data(KBr, cm^{-1}) $\nu_{(OH)}$ 3105, $\nu_{(NH)}$ 3019, $\nu_{(C-O)-}$ 1336, $\nu_{(C=N)}$1531, $\nu_{(N-N)}$ 1015, $\nu_{(C-O-C)}$ 1291, $\nu_{(Sn-O)}$ 695, $\nu_{(Sn-C)}$ 531, $\nu_{(Sn-N)}$ 451; ^{1}H NMR Data (MeOD, δ, ppm) 11.70 (1H, s, OH), 8.30 (1H, s, N=CH). 8.08-6.52 (7H, m, Ar-H), 0.46 (9H, t, J_{Sn-H} = 69Hz, -CH_3); ^{13}C NMR Data (MeOD , δ, ppm) 154.5 (CO=N), 145.4 (CH=N), 145.2, 146.6, 134.9, 128.7, 115.3, 113.2 (Ar-C), 145.5, 143.4, 112.6, 111.9 (furan-C); ^{119}Sn NMR Data(DMSO, δ, ppm) -138.84 ppm.

2.3.2 [(furan-3-ylmethylene)furan-2-carbohydrazide] Trimethyltin(IV) (2)

Quantities used; (furan-3-ylmethylene)furan-2-carbohydrazide (0.204g, 1mmole), KOH (0.1g, 1mmole); $(CH_3)_3SnCl$ (0.2g, 1mmole). Dark brown; Yield: 63% ; Melting point: 180-182^{0}C; Anal. Calcd. for $C_{13}H_{16}N_2O_3Sn$(367): C, 42.55; H, 4.39; N, 7.62%. Found C, 42.57; H, 4.32; N, 7.59%; FT-IR Data (KBr, cm^{-1}) $\nu_{(NH)}$ 3106, $\nu_{(C-O)-}$ 1336, $\nu_{(C=N)}$1530, $\nu_{(N-N)}$ 1014, $\nu_{(C-O-C)}$ 1290, $\nu_{(Sn-O)}$ 689, $\nu_{(Sn-C)}$ 523, $\nu_{(Sn-N)}$ 455; ^{1}H NMR Data(300MHz, MeOD, δ ppm) 8.33 (1H, s, N=CH). 7.94-6.63 (6H, m, Ar-H), 0.411 (9H, t, J_{Sn-H} = 67Hz, -CH_3); ^{13}C NMR Data(δ ppm) 149.8 (CO=N), 147.0 (CH=N), 146.6, 145.6, 138.0, 145.6, 138.0, 115.4, 114.0 (Ar-C).

2.3.3 [(2,4-dichlorobenzylidene)furan-2-carbohydrazide] Trimethyltin(IV) (3)

Quantities used; (2,4-dichlorobenzylidene)furan-2-carbohydrazide (0.17g, 1mmole), KOH (0.1g,1mmole); $(CH_3)_3SnCl$ 0.2g(1mmole). White; Yield: 72% ; Melting point: 155-160^{0}C; Anal. Calcd. for $C_{15}H_{16}N_2Cl_2O_2Sn$(446): C, 40.40; H, 3.62; N, 6.28%. Found C, 40.38; H, 3.66; N, 6.25%; FT-IR Data(KBr, cm^{-1}) $\nu_{(NH)}$ 2921, $\nu_{(C-O)-}$ 1373, $\nu_{(C=N)}$1589, $\nu_{(N-N)}$ 1008, $\nu_{(C-O-C)}$ 1309, $\nu_{(Sn-O)}$ 662, $\nu_{(Sn-C)}$ 515, $\nu_{(Sn-N)}$ 465; ^{1}H NMR Data(MeOD, δ, ppm) 8.83 (1H, s, N=CH). 8.01-6.70 (6H, m, Ar-H), 0.41 (9H, t, J_{Sn-H} = 67Hz, -CH_3); ^{13}C NMR Data(MeOD, δ, ppm) 155.0 (CO=N), 146.6 (CH=N), 143.4, 135.5, 134.3, 131.1, 129.8, 128.5 (Ar-C), 146.6, 143.4, 116.4, 112.6 (furan-C).

2.3.4 [(3-hydroxybenzylidene)furan-2-carbohydrazide] Tribenzyltin(IV) (4)

Quantities used; (3-hydroxybenzylidene)furan-2-carbohydrazide (0.204g, 1mmole), potassium hydroxide (0.1g, 1mmole), $(C_6H_5CH_2)_3SnCl$ (0.43g, 1mmole). Dark yellow; Yield: 55%; Melting point: 113-115^{0}C; Anal. Calcd. for $C_{33}H_{30}N_2O_3Sn$(621) : C, 63.79; H, 4.87; N, 4.51%. Found C, 63.78; H, 4.79; N, 4.53%; FT-IR Data(KBr, cm^{-1}) $\nu_{(OH)}$ 3268, $\nu_{(NH)}$ 3078, $\nu_{(C-O)}$ 1339, $\nu_{(C=N)}$1551, $\nu_{(N-N)}$ 1054, $\nu_{(C-O-C)}$ 1290, $\nu_{(Sn-O)}$ 604, $\nu_{(Sn-C)}$ 501, $\nu_{(Sn-N)}$ 447; ^{1}H NMR Data(300MHz, MeOD, δ ppm), 12.23 (1H, s, OH), 8.65 (1H, s, N=CH). 7.94-6.71 (22H, m, Ar-H), 2.467 (6H, t, J_{Sn-H} = 69Hz, $SnCH_2Ph$); ^{13}C NMR Data(δ ppm) 154.2 (CO=N), 147.0 (CH=N), 132.6, 128.3, 128.0, 124.7, 124.2, 121.4 (Ar-C), 143.3, 139.0, 113.4, 112.8(furan-C).

2.3.5 [((furan-3-ylmethylene)furan-2-carbohydrazide] Tribenzyltin(IV) (5)

Quantities used; (furan-3-ylmethylene)furan-2-carbohydrazide (0.204g, 1mmole), KOH (0.1g, 1mmole), $(C_6H_5CH_2)_3SnCl$ (0.43g, 1mmole); Brown; Yield: 70%; Melting point: 145-148^{0}C; Anal. Calcd. for $C_{31}H_{28}N_2O_3Sn$(595) : C, 62.55; H, 4.74; N, 4.71%. Found C, 62.59; H, 4.72; N, 4.71%; FT-IR Data (KBr, cm^{-1}) $\nu_{(NH)}$ 3105, $\nu_{(C-O)}$ 1386, $\nu_{(C=N)}$1588, $\nu_{(N-N)}$ 1050, $\nu_{(C-O-C)}$ 1291, $\nu_{(Sn-O)}$ 592, $\nu_{(Sn-C)}$ 548, $\nu_{(Sn-N)}$ 450; ^{1}H NMR Data(MeOD, δ, ppm) 8.25 (1H, s, N=CH). 7.76-6.59 (21H, m, Ar-H), 2.53 (6H, t, J_{Sn-H} = 71Hz, $-CH_3$); ^{13}C NMR Data(MeOD, δ, ppm) 149.5 (CO=N), 145.7 (CH=N), 145.1, 139.6, 128.0, 113.7, 111.8 (Ar-C), ^{119}Sn NMR Data(DMSO, δ, ppm) -138.96 ppm.

2.3.5 [(2,4-dichlorobenzylidene)furan-2-carbohydrazide] Tribenzyltin(IV) (6)

Quantities used; (2,4-dichlorobenzylidene)furan-2-carbohydrazide (0.28g, 1mmole), KOH (0.1g, 1mmole); $(CH_3)_3SnCl$ (0.43g, 1mmole), White; Yield: 75%; Melting point: 120-125^{0}C; Anal. Calcd. for $C_{33}H_{28}Cl_2N_2O_2Sn$(674) : C, 58.7; H, 4.19; N, 4.16%. Found C, 58.3; H, 4.13; N, 4.18%; FT-IR Data(KBr, cm^{-1}) $\nu_{(NH)}$ 3132, $\nu_{(C-O)}$ 1374, $\nu_{(C=N)}$1588, $\nu_{(N-N)}$ 1050, $\nu_{(C-O-C)}$ 1291, $\nu_{(Sn-O)}$ 592, $\nu_{(Sn-C)}$ 548, $\nu_{(Sn-N)}$ 450; ^{1}H NMR Data(MeOD, δ, ppm) 8.79 (1H, s, N=CH). 8.26-6.67 (6H, m, Ar-H), 2.52 (6H, t, J_{Sn-H} = 71.4Hz, $SnCH_2Ph$); ^{13}C NMR Data(MeOD, δ, ppm) 145.9 (CO=N), 144.0 (CH=N), 136.4, 129.0, 128.2, 127.6, 127.5, 123.9 (Ar-C), 139.7, 134.7, 116.0, 119.9 (furan-C).

2.3.7 [(3-hydroxybenzylidene)furan-2-carbohydrazide] Triphenyltin(IV) (7)

Quantities used; (3-hydroxybenzylidene)furan-2-carbohydrazide (0.35g, 1.5mmole), potassium hydroxide (0.2g, 1.5mmole), $(C_6H_5)_3SnCl$ (0.58g, 1.5mmole) were used. Brown; Yield: 60% ; Melting point: 105-110^{0}C; Anal. Calcd. for $C_{30}H_{24}N_2O_3Sn$(579) : C, 62.21; H, 4.18; N, 4.84%.

Found C, 62.24; H, 4.23; N, 4.87%; FT-IR Data(KBr, cm^{-1}) $\nu_{(OH)}$ 3268, $\nu_{(NH)}$ 3044, $\nu_{(C-O)-}$ 1330 , $\nu_{(C=N)}$1578, $\nu_{(N-N)}$ 1077, $\nu_{(C-O-C)}$ 1261, $\nu_{(Sn-O)}$ 661, $\nu_{(Sn-C)}$ 454, $\nu_{(Sn-N)}$ 445 ; ^{1}H NMR Data (MeOD, δ, ppm), 11.80 (1H, s, OH), 8.51 (1H, s, N=CH). 7.82-7.41 (22H, m, Ar-H); ^{13}C NMR Data(MeOD, δ, ppm) 152.5 (CO=N), 144.5 (CH=N), 136.0, 129.3, 129.2, 129.1, 128.8(Ar-C), 139.6, 136.5, 128.3, 127.2(furan-C).

2.3.8 [((furan-3-ylmethylene)furan-2-carbohydrazide] Triphenyltin(IV) (8)

Quantities used; (furan-3-ylmethylene)furan-2-carbohydrazide (0.30g, 1.5mmole), KOH (0.2g, 1.5mmole); $(C_6H_5)_3SnCl$ (0.58g, 1.5mmole), Brown; Yield: 65% ; Melting point: 125-128^{0}C; Anal. Calcd. for $C_{28}H_{22}N_2O_3Sn$(553): C, 60.79; H, 4.01; N, 5.06%. Found C, 60.75; H, 3.95; N, 5.04%; FT-IR Data(KBr, cm^{-1}) $\nu_{(NH)}$ 3064, $\nu_{(C-O)-}$ 1291 $\nu_{(C=N)}$1587, $\nu_{(N-N)}$ 1021, $\nu_{(C-O-C)}$ 1291, $\nu_{(Sn-O)}$ 690, $\nu_{(Sn-C)}$ 526, $\nu_{(Sn-N)}$ 462; ^{1}H NMR Data(MeOD, δ, ppm) 8.25 (1H, s, N=CH), 7.72-6.59 (21H, m, Ar-H); ^{13}C NMR Data(MeOD, δ, ppm) 150.0(CO=N), 149.6 (CH=N), 145.6, 145.1, 139.5, 138.6, 113.7, 115.4(furan-C).

2.3.9 [(2,4-dichlorobenzylidene)furan-2-carbohydrazide] Triphenyltin(IV) (9)

Quantities used; (2,4-dichlorobenzylidene)furan-2-carbohydrazide (0.43g, 1.5mmole), KOH (0.2g, 1.5mmole), $(C_6H_5)_3SnCl$ (0.30g,1.5mmole), off white; Yield: 55% ; Melting point: 117-120^{0}C; Anal. Calcd. for $C_{30}H_{22}Cl_2N_2O_2Sn$(632): C, 57.00; H, 3.51; N, 4.43%. Found C, 56.96; H, 3.56; N, 4.40%; FT-IR Data(cm^{-1}) $\nu_{(NH)}$ 3044, $\nu_{(C-O)-}$ 1374 $\nu_{(C=N)}$1589, $\nu_{(N-N)}$ 1018, $\nu_{(C-O-C)}$ 1310, $\nu_{(Sn-O)}$ 616, $\nu_{(Sn-C)}$ 557, $\nu_{(Sn-N)}$ 445; ^{1}H NMR Data(MeOD, δ, ppm) 8.78 (1H, s, N=CH). 8.24-6.66 (21H, m, Ar-H); ^{13}C NMR Data(MeOD, δ, ppm) 156.1 (CO=N), 146.1 (CH=N), 139,4, 136.5, 136.2, 134.7, 129.2, 128.8 (Ar-C), 145.9,144.1, 116.0, 111.9 (furan-C); ^{119}Sn NMR Data(DMSO, δ, ppm) -127.46 ppm.

2.4 X-rays Crystallography

Single crystal analysis for **I_b** was carried out, the crystallographic data and structural refinement details are given in Table 1. During data collection a suitable crystal was selected and kept at 296(2) K. Using Olex2 [26], the structure was resolved with ShelXT structure solution program and structure refinement was done by using Least Squares minimization with ShelXL-1997 refinement package [28].

Table 1 : Crystallographic data and refinement details for (I_b)

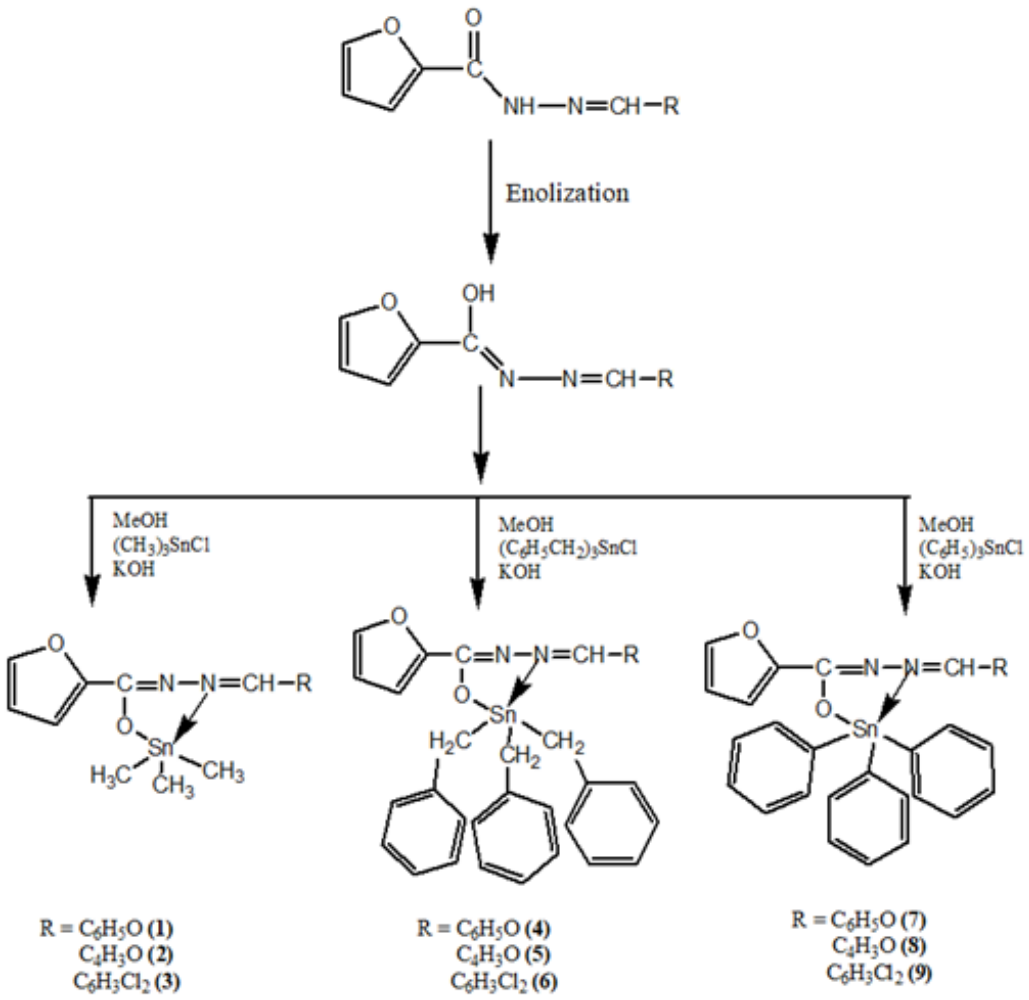
Identification code	**I_b**
Empirical formula	$C_{10}H_8N_2O_3$

Formula weight	204.18
Temperature/K	296(2)
Crystal system	Orthorhombic
Space group	Pbca
a/Å	11.2772(13)
b/Å	7.5345(7)
c/Å	22.812(3)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	1938.3(4)
Z	8
ρ_{calc}/g/cm^3	1.399
μ/mm^{-1}	0.106
F(000)	848.0
Crystal size/mm^3	0.440 × 0.180 × 0.160
Radiation	MoKα (λ = 0.71073)
20 range for data collection/°	5.08 to 53.996
Index ranges	-14 ≤ h ≤ 14, -9 ≤ k ≤ 5, -29 ≤ l ≤ 29
Reflections collected	15332
Independent reflections	2116 [R_{int} = 0.0558, R_{sigma} = 0.0445]
Data/restraints/parameters	2116/0/136
Goodness-of-fit on F^2	1.031
Final R indexes [I>=2σ (I)]	R_1 = 0.0512, wR_2 = 0.1234
Final R indexes [all data]	R_1 = 0.0902, wR_2 = 0.1468
Largest diff. peak/hole / e Å^{-3}	0.20/-0.26

3) Results and Discussion

3.1 Chemistry

All the ligands **(I_a-I_c)** were synthesized using condensation reaction between aromatic aldehydes and 2-furoic acid hydrazide in 1:1 molar ratio to yield hydrazone analogues using the reported methodology[29]. The target compounds **(1-9)** were synthesized by refluxing methanolic solution of respective hydrazone Schiff base with appropriate triorganotin(IV) chloride in the presence of potassium hydroxide, in 1:1 molar ratio (scheme **1)**. These synthesized products are stable in moist air and were fully characterized using sophisticated analytical techniques, the details of which are presented in the experimental section.



Scheme 1

3.2 FT-IR Data

The FT-IR data manifested the presence of all the functionalities found in the target compounds **(1-9)**. Most prominent stretching vibrational band for $\nu_{(C=N)}$ was observed around 1588cm^{-1} in free ligand that was slightly shifted to lower frequency around 1531cm^{-1} on coordination with the metal. Another diagnostic absorption band which aids in determining the binding mode, is the carbonyl stretch $\nu_{(C=O)}$, which appears at 1648cm^{-1} in free ligand and another band for (C-O)$^{-}$ which appears around 1336cm^{-1}. The data clearly manifested that this shift to the lower frequency is due to the coordination of O from carbonyl and N from azomethine moiety with the respective organotin moiety.

Absorption band for $\nu_{(N-N)}$ was observed around 1012 cm^{-1} in free ligand but upon coordination with respective organotin moiety a shift of 5-10 cm^{-1} was observed towards the region of higher

frequency. A band of medium intensity is observed around 1288 cm^{-1} in ligand for ν(C-O-C) of furan ring and it almost remains unaltered in organotin derivatives, which clearly indicate non-involvement of the O belonging to furan ring upon complexation. Metal to ligand vibration bands for Sn-O and Sn-N were observed in far IR region in range of 410-490 cm^{-1} and 470-490 cm^{-1} respectively which provide valid evidence for the formation of complexes.

3.3 ^{1}H, ^{13}C and ^{119}Sn NMR data

The structure of the synthesized complexes were ascertained by using multinuclear (^{1}H, ^{13}C and ^{119}Sn) NMR spectroscopy. The ^{1}H NMR data explicitly determine all the magnetically equivalent protons present in the ligand, as well as in the targeted compounds **(1-9)**. In case of triorganotin(IV) complexes ^{n}J[Sn-^{1}H] coupling values further assist in determining geometric shape of the complex[30]. The chemical shift value for **N-H** proton appear in range 9.65-11.82ppm belong to hydrazone Schiff base where as it disappeared in the spectra of all the complexes. It provides evidence that complexation occur through N-H functionality of the ligand. The non-involvement of –OH proton in complexation can be inferred from the resonance occurred at about 11.81ppm. All the chemical shift values for furan and phenyl ring protons appears in their usual regions both for the precursors and targeted compounds. Some additional satellite peaks were also observed for trimethyltin(IV) derivatives at 0.46(**1**), 0.41(**2**), 0.41(**3**)ppm which refers to ^{2}J [^{119}Sn, ^{1}H].

The coupling constant ^{2}J [^{119}Sn, ^{1}H] for **(1)** a representative trimethyltin(IV) derivative **1** was calculated and found to be 69Hz. Whereas θ value is to be 119° by using Lockhart-Manders equation[31]. The coupling constant ^{2}J [^{119}Sn, ^{1}H] for a representative tribenzyltin(IV) derivative **6** was calculated and found to be 71Hz. Whereas θ value was determined to be 121° and all these calculated values are well documented that clearly demonstrated that all triorganotin(IV) derivatives are five coordinated having trigonal bipyramidal as their geometric shape [32, 33].

The ^{13}C NMR data clearly resolved all the unique carbons for **(1-9)**. The two characteristics resonances pertaining to **C=O** and **–N=CH** moieties appear at 165.3 and 154.5 ppm respectively, these signals are slightly shifted to upfield regions for all the complexes. The aromatic carbons appear in their usual region. Furthermore characteristic resonances for all the complexes were also observed and are given in the experimental section.

The ^{119}Sn NMR spectroscopy is another diagnostic tool to determine the geometry and coordination number of synthesized organotin derivatives. The chemical shift values for **1**, **5** and

9 lie in range -90 to -190ppm which is the reference value for five coordinated organotin compound[34-36]. This data provide evidence that the geometry of all the synthesized complexes is distorted trigonal bipyramidal[32].

3.4 X-ray Crystallography

ORTEP diagram is delineated in Fig.1. The structural refinement parameters and crystallographic data for **(I_b)** are presented in Table 1. The data for selected bond lengths, bond angles and hydrogen bonding have been given as supplementary information. The data suggest that eight molecules of **(I_b)** are enclosed in one unit cell. The bond distance for C(5)- O(2) and C(6) -N(2) is 1.231(2) and 1.273(2)˚A respectively which is in close agreement with the reported bond length for double bond[27]. The N(1)- N(3) bond distance is 1.377(2)˚A which is shorter than the bond distance of single bond that is, 1.411(7)˚A showing some double bond character[27]. Bond angles for O(2)- C(5)- N(1) and O(2)- C(5)- C(4) are 123.03˚(18) and 120.27˚(18) respectively, exhibiting a planer structure for the molecule.

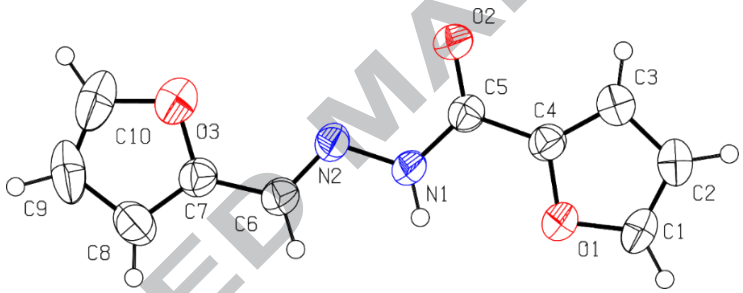


Figure 1. ORTEP diagram for I_b

3.5 Biological studies

Triorganotin(IV) complexes are potentially bioactive owning to their biocidal properties and are used as an industrial biocides like fungicides, insecticide and also as a rodent repellent. The following biological activities were carried out to further explore their bioeffectiveness.

3.5.1 Antibacterial activity

All the synthesized compounds were preliminarily screened for their antibacterial potential. The compounds **1**, **5**, **7**, **8** and **9** exhibited good zone of inhibitions against *P. aeruginosa*. whereas **4-6** were found to exhibit moderate zones (13 mm) of inhibition against *B. subtilis*, *K. pneumoniae* and *E. coli* and **7-9** specifically displayed 12–17 mm zone of inhibition against *B. subtilis* and *E. coli* in comparison to the positive control. Owing to spatial conformations the triorganotin compounds gain entry into the cell lines of an organism, thus inhibiting the bacterial growth considerably. Here zone of inhibition ≥ 13 mm in diameter was considered active and was

further screened for MIC values on ninety six well plate. After incubation of 24hours, the minimum bactericidal concentration MBC was evaluated only for those compounds that show clear wells, demonstrating that the compound arrested the bacterial growth within this period of time. The MBC results demonstrate bacteriostatic nature of the compounds such as **1**, **5**, **7** and **8** against *P. aeruginosa*, **4** against resistant *S. haemolyticus* and **9** against *E. coli*[37].

Table 2 : Antibacterial Activity data[ab] for (1-9)

Comp. No.	*S. aureus*		*B. subtilis*		*K. pneumoniae*		*E. coli*		*P. aeruginosa*		Resistant *E. coli*		Methicillin Resistant *S. aureus*		R *h*
	ZOI (mm)	MIC mM (µg/mL)	ZOI (mm)	MIC mM (µg/mL)	ZOI (mm)	MIC mM (µg/mL)	ZOI (mm)	MIC mM (µg/mL)	ZOI (mm)	MIC mM (µg/mL)	ZOI (mm)	MIC mM (µg/mL)	ZOI (mm)	MIC mM (µg/mL)	Z (
Ia	---	---	---	---	9.33± 0.58	0.216 (50)	---	---	---	---	---	---	---	---	--
Ib	---	---	---	---	7.33± 0.58	0.244 (50)	---	---	---	---	---	---	8.33± 0.58	0.244 (50)	--
Ic	---	---	---	---	7.33± 0.58	0.177 (50)	---	---	---	---	---	---	13.33 ±0.58	0.177 (50)	--
1	---	---	8.33± 0.58	0.127 (50)	9.33± 0.58	0.127 (50)	---	---	---	---	---	---	---	---	--
2	---	---	9.33± 0.58	0.136 (50)	9.33± 0.58	0.136 (50)	---	---	15.33 ±0.58	0.136 (50)	7.33± 0.58	---	13.33 ±0.58	0.136 (50)	1 ±
3	---	---	---	---	6.33± 0.58	---	---	---	6.33± 0.58	---	---	---	---	---	1 ±
4	---	---	12.33 ±0.58	0.080 (50)	12.33 ±0.58	0.080 (50)	12.33 ±0.58	0.080 (50)	---	---	---	---	---	---	--
5	7.33± 0.58	0.084 (50)	12.33 ±0.58	0.084 (50)	12.33 ±0.58	0.084 (50)	12.33 ±0.58	0.084 (50)	11.33 ±0.58	0.084 (50)	16.33 ±0.58	0.084 (50)	12.33 ±0.58	0.084 (50)	1 ±
6	---	---	12.33 ±0.58	0.074 (50)	12.33 ±0.58	0.074 (50)	12.33 ±0.58	0.074 (50)	14.33 ±0.58	0.0027 (1.85)	13.33 ±0.58	0.074 (50)	18.33 ±0.58	0.074 (50)	1 ±
7	---	---	11.33 ±0.58	0.086 (50)	9.33± 0.58	0.086 (50)	13.33 ±0.58	0.086 (50)	---	---	6.33± 0.58	---	---	---	--
8	---	---	13.33 ±0.58	0.090 (50)	10.33 ±0.58	0.090 (50)	12.33 ±0.58	0.090 (50)	14.33 ±0.58	0.0033 (1.85)	12.33 ±0.58	0.090 (50)	13.33 ±0.58	0.090 (50)	1 ±
9	---	---	12.33 ±0.58	0.079 (50)	10.33 ±0.58	0.079 (50)	16.33 ±0.58	0.026 (16.6)	15.33 ±0.58	0.0029 (1.85)	10.33 ±0.58	0.079 (50)	11.33 ±0.58	0.079 (50)	1 ±
cefixime	24.33 ±0.58	0.00055 (0.25)	29.33 ±0.58	0.00017 (0.08)	24.33 ±0.58	0.00055 (0.25)	13.33 ±0.58	0.00055 (0.25)	13.33 ±0.58	0.044 (20)	13.33 ±0.58	0.044 (20)	10.33 ±0.58	0.044 (20)	1 ±

a) Values are presented as mean ± standard deviation of triplicate analysis.

b) --- : Indicates bacterial strain is resistant to the compound at >50 µg/ml.

3.5.2 Antifungal activity

All the synthesized compounds were also screened for antifungal activity and the data manifested moderate zone of inhibition in range 12-20 mm against all the tested strains for **7-9**, whereas **1** and **2** were specifically more active against *F. solani* in range 15-20 mm. The compound **7**, **8** and **9** exhibited good activity against all the fungal strains as compare to clotrimazole and their MIC values fall in range 6.25-1.56 µg/ml. The data suggest that the activity of the compounds are mainly governed by the nature of triorganotin moiety, and play a pivotal role in inhibiting the growth of the tested organisms. The differential behavior of compounds in terms of specificity was further determined by varying the substituents attached to the respective organotin moiety. The fugicidal activity of triphenyltin derivatives was significant followed by trimethyl and tribenzyltin derivatives [38].

Table : 3 Antifungal Activity data[ab] for (1-9)

No. of compound	*Mucor sp.*	MIC mM (µg/mL)	*F. solani*	MIC mM (µg/mL)	*A. niger*	MIC mM (µg/mL)	*A. fumigatus*	MIC mM (µg/mL)	*A. flavus*
Ia	---	---	---	---	---	---	---	---	10.33±0.58
Ib	---	---	6.33±0.58	0.244 (50)	---	---	---	---	8.33±0.58
Ic	---	---	7.33±0.58	0.177 (50)	---	---	---	---	8.33±0.58
1	11.33±0.58	0.127 (50)	14.33±0.58	0.031 (12.5)	8.33±0.58	0.127 (50)	---	---	8.33±0.58
2	13.33±0.58	0.017 (6.25)	19.33±0.58	0.034 (12.5)	9.33±0.58	0.136 (50)	11.33±0.58	0.136 (50)	9.33±0.58
3	---	---	8.33±0.58	0.112 (50)	---	---	---	---	14.33±0.58
4	7.33±0.58	0.080 (50)	8.33±0.58	0.080 (50)	---	---	7.33±0.58	0.080 (50)	12.33±0.58
5	7.33±0.58	0.084 (50)	8.33±0.58	0.084 (50)	6.33±0.58	0.084 (50)	7.33±0.58	0.084 (50)	9.33±0.58
6	7.33±0.58	0.074 (50)	8.33±0.58	0.074 (50)	6.33±0.58	0.074 (50)	9.33±0.58	0.074 (50)	8.33±0.58
7	13.33±0.58	0.0026 (1.56)	14.33±0.58	0.021 (12.5)	15.33±0.58	0.0026 (1.56)	12.33±0.58	0.0026 (1.56)	18.33±0.58
8	14.33±0.58	0.009 (6.25)	16.33±0.58	0.009 (6.25)	15.33±0.58	0.002 (1.56)	13.33±0.58	0.002 (1.56)	20.33±0.58
9	13.33±0.58	0.011 (6.25)	15.33±0.58	0.011 (6.25)	14.33±0.58	0.002 (1.56)	12.33±0.58	0.002 (1.56)	18.33±0.58
clotrimazole	29.33±0.58	0.014 (5)	16.33±0.58	0.014 (5)	21.33±0.58	0.014 (5)	22.33±0.58	0.014 (5)	31.33±0.58

a) Values are presented as mean ± standard deviation of triplicate analysis.

b) --- : Indicates fungal strain is resistant to the compound at >50 µg/ml.

3.5.2 Antidiabetic Activity (alpha amylase inhibition assay)

Alpha amylase inhibition assay was performed to determine antidiabetic activity for **(1-9)**. It may be inferred from Fig. 2 that moderate biological activity was observed for **7-9** in range 35- 40% at concentration 0.086, 0.090 and 0.079mM, whereas **4-6** demonstrate 20-25% enzyme inhibitions at 0.080, 0.084 and 0.074mM as compare to acarbose. The data clearly demonstrated that the compounds are moderately effective alpha amylase inhibitor, however further research may be carried out in this domain to arrive at a fruitful conclusion. Inspite of that the antidiabetic activity for the present regime varied in the following order; triphenyltins > tribenzyltins >trimethyltins.

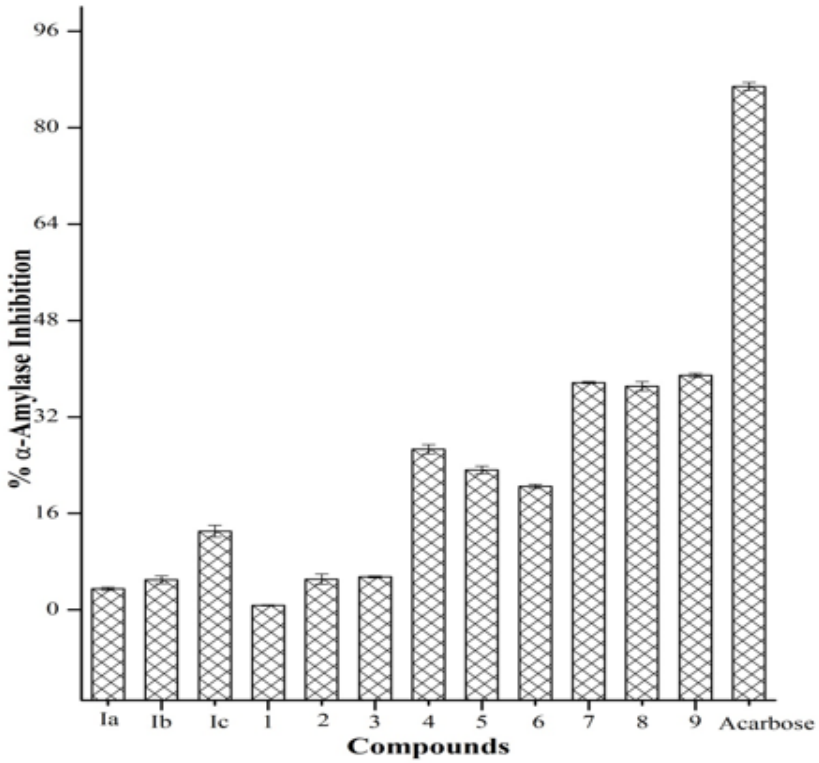


Figure 2. α-amylase inhibition assay for (1-9).

3.5.3 Free Radical Scavenging Activity

The DPPH activity data have been depicted in Fig.3 described that almost all the synthesized ligands and organotin(IV) derivatives show good activity with the exception of triphenyltin derivatives which exhibit poor activity in comparison to positive control. The compounds **4** and **5** showed highest percentage scavenging activity of 31.63% and 18.44% at concentration of 0.322 and 0.336 mM. It has been argued that tribenzyltin(IV) derivatives, being electron donor in nature, stabilize DPPH radical more effectively that reduces it to exhibit more scavenging

activity as compare to triphenyltin(IV) derivatives**(7-9)** which were tested at the concentration of 0.345, 0.361 and 0.316 mM respectively.

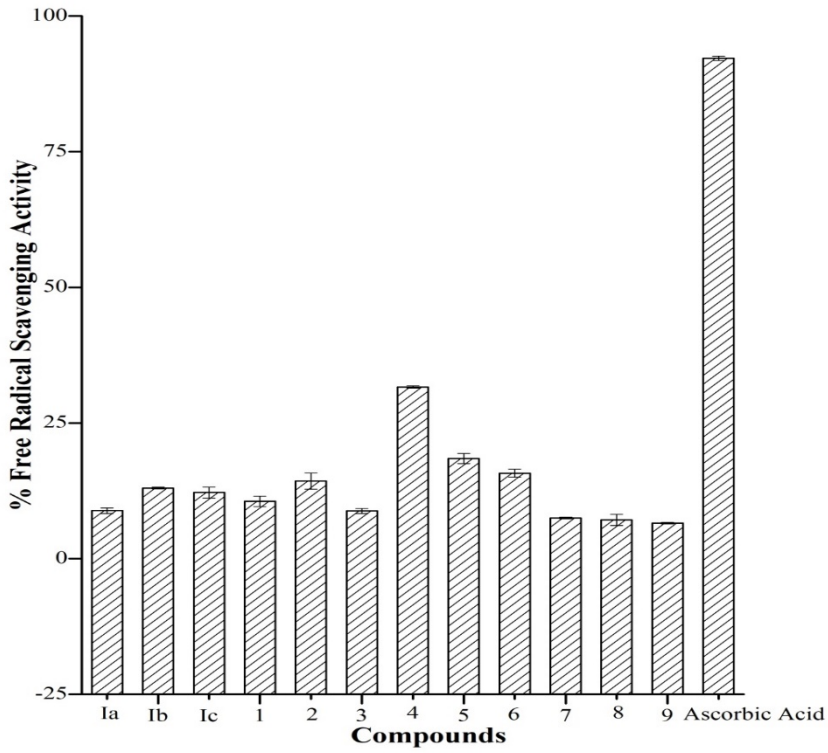


Figure 3 % Free radical scavenging activity for compounds (1-9).

3.5.4 Total Antioxidant Activity

The antioxidant mechanism involves electron transfer which is dependent on the structure of antioxidant agent. The compounds **2**, **5** and **6** (which were tested at 0.544, 0.336 and 0.296 mM concentrations respectively) exhibited the highest antioxidant capacity (with TAC values of 55.70 ± 0.77 and 54.57 ± 1.02 µg AAE/mg respectively). The data suggest that they may find themselves as promising antioxidant agents in future drug development strategy.

3.5.5 Total Reducing Power Assay

The ferric reducing power assay is yet another method to determine the reducing power of the synthesized compounds involving conversion of potassium ferricyanide to potassium ferrocyanide that gives intense blue colored complex[39]. The compounds **2**, **5** and **6** demonstrated highest reducing potential with TRP values of 387.42 ± 0.75, 307.97 ± 0.79 and

321.89 ± 0.83 µg AAE/mg respectively at 0.544, 0.336 and 0.296 mM. whereas **I_b** exhibit TRP value of 183.98±1.25µg AAE/mg at 0.980 mM concentration.

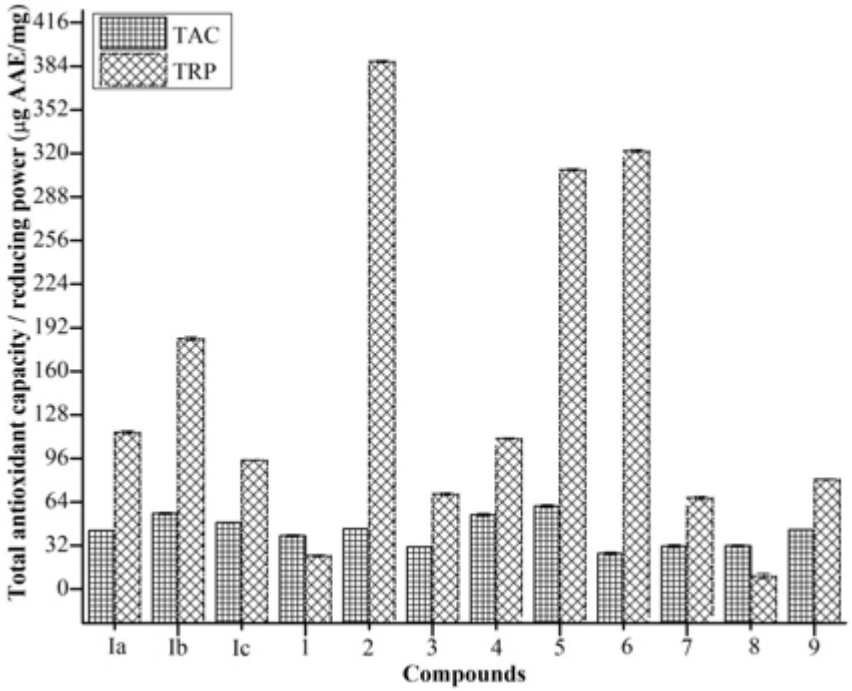


Figure 4: Total antioxidant capacity and total reducing power for (1-9).

3.5.6 Cytotoxicity (brineshrimp lethality assay)

The brine shrimp lethality assay is a simple, robust and inexpensive method for the assessment of toxicity of any natural or synthetic product. High lethality with LC_{50} < 100 µg/ml has been measured by brineshrimp lethality assay that may be categorized as toxic. Highest toxicity with LC_{50} < 30 µg/ml has been assigned to **2**, **3**, **4** and **8** whereas **3** showed LC_{50} value of 19.76 µg/ml at concentration of 0.448mM and **8** show LC_{50} value of 16.62 µg/ml at a low concentration of 0.316mM demonstrated even more toxic. The data suggest that the synthesized organotin(IV) derivatives are more toxic owing to inherited toxicity of these organomettalics, whereas some modifications are needed to develop a compromise between the toxicity and activity of such kind of compounds for future drug discovery processes.

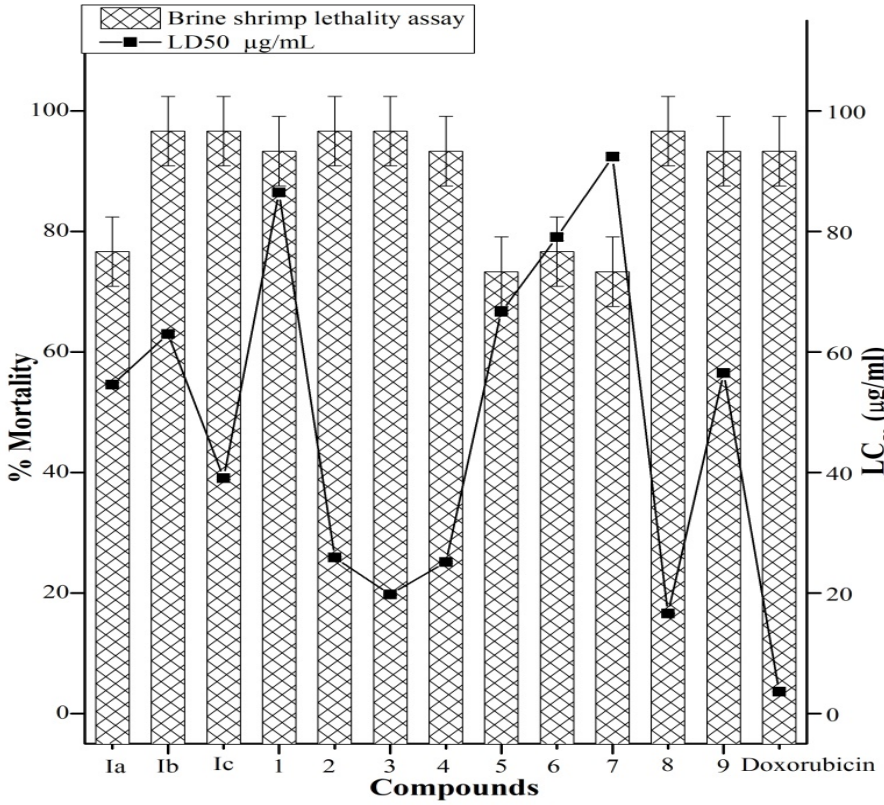


Figure 5 Brine shrimp lethality assay for (1-9).

4) Conclusions

Three new hydrazone Schiff bases **(Ia-Ib)** and their organotin(IV) derivatives **(1-9)** were successfully synthesized and well characterized by using FT-IR, multinuclear (^{1}H, ^{13}C, ^{119}Sn) NMR spectroscopy and single crystal X-ray diffraction analysis. All the synthesized compounds were also evaluated to check their biological significance for development of limited structure-activity relationship and some generalization has been drawn. The data suggest that tribenzyl and triphenyltin derivatives exhibit good antibacterial and antifungal activity whereas compounds **2**, **5** and **6** show excellent results vis-a-vis total reducing power assay.

Acknowledgements

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Appendix A. Supplementary data

The CCDC for the reported compound was 1837071. It can be free download from CCDC data base. These data can be obtained free of charge via

http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

Abbreviations

DPPH : 2, 2-Diphenyl-1-picrylhydrazyl, **FRSA**: Free radical scavenging activity, **IC_{50}** : 50% Inhibitory concentration, **TAC**: Total antioxidant capacity, **TRP**: Total reducing power, **DMSO** : Dimethyl sulfoxide, **AAE** : Ascorbic acid equivalent, **MIC** : Minimum inhibitory concentration, **MBC** : Minimum bactericidal concentration.

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- Some new substituted hydrazones have been synthesized as precursors.
- These precursors then complexed with various triorganotin compounds to further explore their structural as well as biological aspects.
- All these compounds were fully characterized by advanced analytical techniques for their structural validation.
- All these compounds were preliminary screened to evaluate their various bioactivities and found some encouraging results.

Synthetic Stratagem, Characterization and Biocidal applications of Triorganotin(IV) Complexes derived from Hydrazide/Hydrazone Analogues

Mehwish Mehmood[a], Imtiaz-ud-Din[a*], Muhammad Nawaz Tahir[b], Ihsan-ul Haq[c], Syeda Sania Zahra[c]

Graphical Abstract: Synopsis

A series of new triorganotin(IV) complexes of Schiff base ligands as benzylidene benzohydrazide and their hydrazone analogues have been synthesized. Then they were complexed with organotin(IV) moiety with general formula [R'_3SnL] where L= C_4H_3O-CONH-N=CH-R, R= C_6H_5O(**I_a**), C_4H_5O(**I_b**), $C_6H_3Cl_2$(**I_c**) and R'= -CH_3(**1-3**), -CH_2Ph(**4-6**), -Ph(**7-9**). The complexes **(1-9)** have been fully characterized by IR, NMR(^1H, ^{13}C and ^{119}Sn) spectroscopy, combined with CHN and melting point determinations.