Research Article

# Synthesis, Characterization and *In-Vitro* Biological Assays of Triphenyltin Derivatives of Phenylhydrazones

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**ABSTRACT**

Schiff base formation is a very important reaction of pharmaceutical chemistry. Schiff bases are gaining importance day by day as they have exhibited useful biological importance and are found to be a versatile pharmacophore for design and development of many bioactive compounds**.** Schiff's bases of Phenyl hydrazine with the benzaldehyde, salicylaldehyde, cinnamonaldehyde, acetophenone and 2-hydroxyacetophenone were synthesized by condensation method. After the formation of the Schiff's bases their Triphenyltin derivatives were also synthesized. Spectroscopic characterization of all these Schiff's bases and their tin derivatives were done through FT-IR, $^{1}$H NMR and $^{13}$C NMR. These Schiff's bases and their complexes were screened through in-vitro antibacterial and antifungal activities and some of the triphenyltin derivatives were found to have impressive results. Moreover further investigation regarding pharmacological activities of the synthesized Schiff's bases and their derivatives are a part of our future research plan.

**Keywords:** Biological Assays, Phenylhydrazine, Schiff's Bases, Triphenyltin complexes.

## INTRODUCTION

Compounds containing azomethine group (-CH=N-) are known as Schiff's bases and they are synthesized by the condensation of primary amine with the carbonyl compounds (aldehyde and ketones).[1] Schiff's bases have great significance in medicinal chemistry.[2] Schiff's bases are the important compound owing to their wide range of biological activities and industrial application.[3] They have been found to possess the pharmacological activities such as antimalarial[4], anticancer[5], antibacterial[6], antifungal[7], antiviral[8] and anti-inflammatory.[9,10]

The biological activity and chemistry of Schiff's bases and their transition metal complexes showed notable interest in the past.[11-14] Schiff's bases play worth mentioning role in inorganic chemistry as they form stable complexes with most transition metal ions.[15,16] Metal complexes of Schiff's bases also have industrial, antifungal, antibacterial, anticancer and herbicidal applications.[17,18]

Organotin (IV) compounds, a deceptively simple area of inorganic and organometallic chemistry, have been receiving more attention due to their industrial, herbicidal, medicinal and environmental applications.[19,20] Nitrogen, oxygen, and sulphur donor ligands have been used to enhance the biological activity of organotin derivatives.[21] Present study highlights the synthesis of the Schiff's bases and their triphenyltin complexes with their spectroscopic characterization followed by their In-vitro antibacterial and antifungal assays. During the screening process some of the triphenyltin complexes of the Schiff's bases of phenyl hydrazine were found to be very active with impressive results.

## MATERIALS AND METHODS

Triphenyltin chloride, Benzaldehyde, Salicylaldehyde Cinnamonaldehyde, Acetophenone, Phenyl hydrazine, 2-Hydroxyacetophenone, Ethanol and Methanol were purchased from Aldrich and were used without any further purification. Anhydrous toluene was obtained by drying with sodium metal wire. Fourier Transform Infrared spectra of the Schiff's bases and their metal complexes were recorded directly on Bruker FTIR spectrometer. $^{1}$H and $^{13}$C NMR spectra were measured on a Bruker 400MH spectrometer (Germany). Melting points were determined by using a Gallenkamp melting point apparatus. All the glass apparatuses were dried at 120°C throughout the experimental work.

### General Procedure for Synthesis of Schiff's bases

These Schiff's bases were synthesized by the condensation of phenyl hydrazine with one to one molar ratio with redistilled different aromatic aldehydes and ketones. The reaction mixture refluxed for one hour. Ethanol was used as a solvent and acetic acid as a catalyst. The reaction mixture filtered after cooling and products were recrystallized with ethanol.[22]

### Synthesis of Triphenyltin Complexes of Schiff's Bases of Phenyl hydrazine

A solution of Schiff's bases of phenyl hydrazine ($LH^{6}$ – $LH^{10}$) (1mmole) is suspended in dry toluene (50 ml) and treated with triethylamine $Et_3N$ (1mmole). The mixture was refluxed for 3 hours. In dry toluene triphenyltin chloride (1mmole) was added as solid to a reaction flask with constant stirring and the reaction mixture refluxed for 3h. The reaction mixture contains $Et_3NHCl$ is filtered off such that filtrate had the organotin derivative. The

solvent was removed through rotary apparatus. The mass left behind will be recrystallized from $CHCl_3$.[23-25]

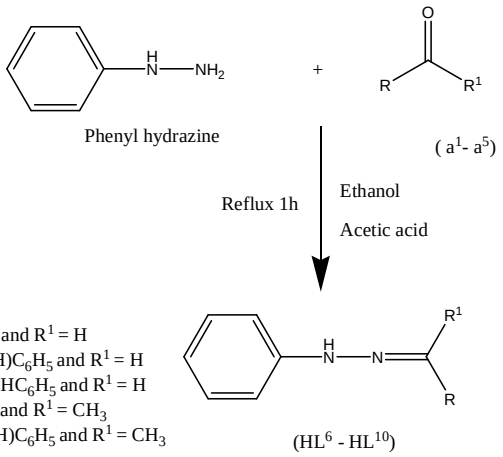


**Figure 1:** Synthetic scheme of Phenyl hydrazine Schiff's bases

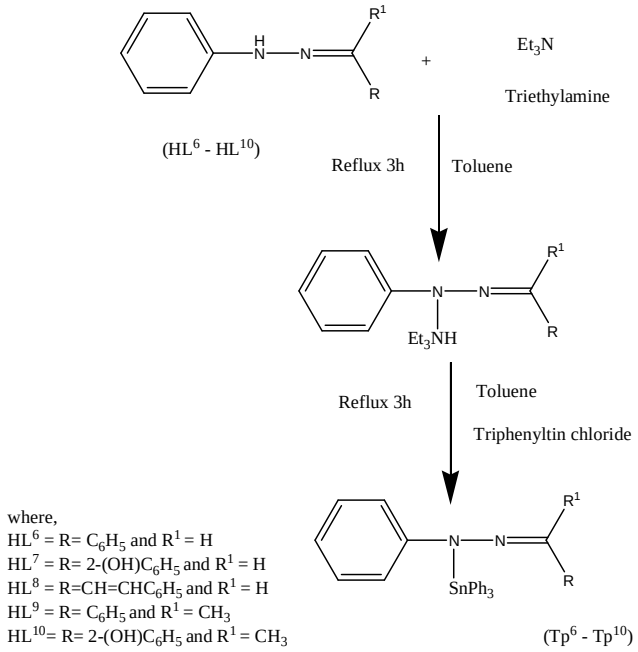


**Figure 2:** Synthetic scheme of Triphenyltin Complexes of Phenyl hydrazine Schiff's Bases

All the Schiff's Bases and their derivatives were synthesized with the procedure mentioned above and their physical and chemical characteristics are given below.

### Schiff's Base HL[6] (Schiff's base of Phenyl hydrazine with Benzaldehyde)

Yield (58 %), m. p. 150 °C, Elemental Analysis: calculated for $C_{13}H_{12}N_2$: C, 79.56 ; H, 6.16; N, 14.27; found: C, 79.60; H, 6.10; N, 14.30; FT-IR (4000-400 $cm^{-1}$) 3320 ν(NH), 3030ν(CH), 1655 ν(C=N), ν 1490 (CH=CH),1132 ν(C–N), $^1$H NMR ($DMSO_{4-D6,}$ ppm), 7.51-7.55d; 7.3-7.34m; 7.25-7.28m (–$C_6H_5$), 6.72-7.76d; 7.01-7.05m; 6.8m (NH–$C_6H_5$), 7.73s (=CH–), 3.5s (–NH), $^{13}$C NMR ($DMSO_4$–D6, ppm), 144.6(C-1), 112.65 (C-2/5), 129.2 (C-3/4), 112.7 (C-6), 137.3 (C-7), 137.21 (C-8), 128.6 (C-9/13), 128.3 (C-10/11), 135.21 (C-12)

### Schiff's Base HL[7] (Schiff's base of Phenyl hydrazine with salicylaldehyde)

Yield ( 62%), m. p. 170 °C, Elemental Analysis: calculated for $C_{13}H_{12}N_2O$: C, 73.56; H, 5.70; N, 13.20; found: C, 73.59; H, 5.59; N, 13.23; FT-IR (4000-400 $cm^{-1}$) 3289 ν(NH), 3053ν(CH), 1622 ν(C=N), 1496 ν (CH=CH), 1148 ν(C–N), 1253 ν(C–O) $^1$H NMR ($DMSO_{4-D6,}$ ppm), 7.26-7.31d; 7.21-7.25m; 7.17-7.19d; (–$C_6H_5OH$), 6.71-6.76d; 7.00-7.05m; 6.78-6.81m (NH–$C_6H_5$), 7.74s (=CH–), 4.5s (–NH–), 5.6s (–OH), $^{13}$C NMR ($DMSO_4$–D6, ppm), 143.2(C-1), 116.53 (C-2/5), 129.5 (C-3/4), 119.53 (C-6), 141.09 (C-7), 118.46 (C-8), 159.43 (C-9/13), 112.56 (C-10/11), 129.98 (C-12).

### Schiff's Base HL[8] (Schiff's base of Phenyl hydrazine with Cinnamonaldehyde)

Yield (48%), m. p. 140°C, Elemental Analysis: calculated for $C_{15}H_{14}N_2$: C, 81.05; H, 6.35; N, 12.60; found: C, 81.03; H, 6.42; N, 12.55; FT-IR (4000-400 $cm^{-1}$) 3312ν(NH), 3040 ν(CH), 1670 ν(C=N), 1486 ν (CH=CH), 1135 ν(C–N), $^1$H NMR ($DMSO_{4-D6,}$ ppm), 7.33-7.35d; 7.17-7.20m; 7.12-7.15m (–$C_6H_5$), 6.58-6.62d; 6.80-6.82m; 6.66m (NH–$C_6H_5$), 7.4s (=CH–), 7.1s (–NH–), 5.27m; 6.7d (–CH=CH–), $^{13}$C NMR ($DMSO_4$–D6, ppm), 144.25 (C-1), 112.67 (C-2/5), 129.29 (C-3/4), 120.15 (C-6), 136.7 (C-7), 125.87 (C-8), 139.37 (C-9), 134.04 (C-10), 126.51 (C-11/15), 128.7 (C-12/14), 128.47 (C-13).

### Schiff's Base HL[9] (Schiff's base of Phenyl hydrazine with Acetophenone)

Yield ( 71%), Semisolid, Elemental Analysis: calculated for $C_{14}H_{14}N_2$: C, 79.97; H, 6.71; N, 13.32; found: C, 79.84; H, 6.79; N, 13.37; FT-IR (4000-400 $cm^{-1}$) 3300 ν(NH), 3058 ν(CH), 1673 ν(C=N), 1492 ν (CH=CH), 1072 ν(C–N), $^1$H NMR ($DMSO_{4-D6,}$ ppm), 7.90-7.95d; 7.41-7.45m; 7.22m; (–$C_6H_5$), 6.52-6.55d; 7.13-7.15m; 668m (NH–$C_6H_5$), 7.22s (–NH–), 1.5s (–$CH_3$), $^{13}$C NMR ($DMSO_4$–D6, ppm), 141.8 (C-1), 114.5 (C-2/5), 128.54 (C-3/4), 118.51 (C-6), 25.5 (C-7), 151.25 (C-8), 132.12 (C-9), 128.28 (C-10/14), 127.71 (C-11/13), 128.63 (C-12).

### Schiff's Base HL[10] (Schiff's base of Phenyl hydrazine with 2-Hydroxyacetophenon)

Yield ( 65%), m. p. 107°C, Elemental Analysis: calculated for $C_{14}H_{14}N_2O$: C, 74.31; H, 6.24; N, 12.38; found: C, 74.20; H, 6.32; N, 12.34; FT-IR (4000-400 $cm^{-1}$) 3354 ν(NH), 3070 ν(CH), 1640 ν(C=N), 1495 ν (CH=CH), 1173 ν(C–N), $^1$H NMR ($DMSO_{4-D6,}$ ppm), 7.35-7.39d; 7.28-7.30m; 7.24-7.26m (–$C_6H_5OH$), 6.80-6.82d; 7.19-7.22m; 6.92m (NH–$C_6H_5$), 7.01s (–NH–), 2.27s(–$CH_3$), 3.8(–OH), $^{13}$C NMR ($DMSO_4$–D6, ppm), 143.85(C-1), 117.3 (C-2/5), 129.51 (C-3/4), 118.8 (C-6), 11.64 (C-7), 157.88 (C-8), 119.2 (C-9), 147.39 (C-10/14), 129.91 (C-11/13), 129.75 (C-12).

### Tp[6] (Triphenyltin derivative of Schiff's base HL[6])

Yield ( 35%), m. p. 120°C, Elemental Analysis: calculated for $C_{31}H_{26}N_2Sn$: C, 64.84; H, 4.39; N, 7.32; found: C, 64.79; H, 4.24; N, 7.45; FT-IR (4000-400 $cm^{-1}$) 3020 ν(CH), 1689 ν(C=N), 1479 ν(CH=CH), 1152ν(C–N), 443ν(Sn–N) $^1$H NMR

($DMSO_{4-D6}$, ppm), 8.47-8.5d; 7.89-7.91m; 7.85m (–$C_6H_5$), 7.41-7.43d; 7.48-7.50m; 7.46m (NH–$C_6H_5$), 8.8s (=CH–), 7.34-7.40m, 7.30m ($C_6H_5$)Sn), $^{13}$C NMR ($DMSO_4$–D6, ppm), 148.99(C-1), 121.53 (C-2/5), 128.9 (C-3/4), 127.2 (C-6), 136.3 (C-7), 133.93 (C-8), 128.73(C-9/13), 128.5 (C-10/11), 130.40 (C-12), 129.8 (C-14), 136.8 (C-15/19), 128.3 (C-16/18), 128.13 (C-17).

### Tp[7] (Triphenyltin derivative of Schiff's base HL[7])

Yield (49 %), m. p. 130°C, Elemental Analysis: calculated for $C_{31}H_{26}N_2OSn$: C, 66.34; H, 4.67; N, 4.99; found: C, 66.40; H, 4.69; N, 4.91; FT-IR (4000-400 $cm^{-1}$) 3070 ν(CH), 1620 ν(C=N), 1492 ν(CH=CH), 1146 ν(C–N), 1253 ν(C–O), 442 ν(Sn–N), $^1$H NMR ($DMSO_{4-D6}$, ppm), 7.44-7.47m; 6.85-6.88m; 7.18d (–$C_6H_5OH$), 6.75-6.77d; 6.95-6.99m; 6.8m (N–$C_6H_5$), 8.2s (=CH–), 4.6s (–OH), 7.28m (Sn–$C_6H_5$), $^{13}$C NMR ($DMSO_4$–D6, ppm), 144.7 (C-1), 115.84 (C-2/5), 129.17 (C-3/4), 119.31 (C-6), 137.01 (C-7), 118.91 (C-8), 155.57 (C-9/13), 111.64 (C-10/11), 129.27 (C-12), 120.48 (C-14), 136.2 (C-15/19), 129 (C-16/18), 128.3 (C-17).

### Tp[8] (Triphenyltin derivative of Schiff's base HL[8])

Yield ( 57 %), m. p. 125°C, Elemental Analysis: calculated for $C_{33}H_{28}N_2Sn$: C, 69.38; H, 4.94; N, 4.90; found: C, 69.45; H, 4.89; N, 4.99; FT-IR (4000-400 $cm^{-1}$) 3030 ν(CH), 1680ν(C=N), 1487 ν(CH=CH), 1136 ν(C–N), 444 ν(Sn–N) $^1$H NMR ($DMSO_{4-D6}$, ppm), 7.32-7.35d; 7.20-7.22m; 7.16-7.18m (–$C_6H_5$), 6.69-6.71d; 7.0-7.02m; 6.78m (NH–$C_6H_5$), 7.89s (=CH–), 5.21m; 5.9d (–CH=CH–), 7.33 (Sn-$C_6H_5$) $^{13}$C NMR ($DMSO_4$–D6, ppm), 144.89 (C-1), 111.85 (C-2/5), 129.09 (C-3/4), 118.73 (C-6), 136.14 (C-7), 126.36 (C-8), 139.26 (C-9), 132.8 (C-10), 126.46 (C-11/15), 128.7 (C-12/14), 127.6 (C-13), 136.7 (C-16), 126.8 (C-17/21),129 (C-18/20), 128.5 (C-19).

### Tp[9] (Triphenyltin derivative of Schiff's base HL[9])

Yield (61%), m. p. 190°C, Elemental Analysis: calculated for $C_{32}H_{28}N_2Sn$: C, 68.72; H, 5.05; N, 5.01; found: C, 68.60; H, 5.12; N, 5.20; FT-IR (4000-400 $cm^{-1}$) 3030 ν(CH), 1678 ν(C=N), 1479 ν(CH=CH), 1073ν(C–N), 453 ν(Sn–N), $^1$H NMR ($DMSO_{4-D6}$, ppm), 7.89-7.92d; 7.58-7.62m; 7.40m (–$C_6H_5$), 6.67-6.7d; 7.16-7.18m; 6.84m (N–$C_6H_5$), 1.12s ($CH_3$–), 7.32m (Sn-$C_6H_5$), $^{13}$C NMR ($DMSO_4$–D6, ppm), 142.5 (C-1), 114.23 (C-2/5), 128.9 (C-3/4), 117.6 (C-6), 22.3 (C-7), 152.3 (C-8), 131.75 (C-9), 128.11 (C-10/14), 127.37 (C-11/13), 128.54 (C-12), 129.3 (C-15), 136.3 (C-16/20), 128.8 (C-17/19), 128.3 (C-18).

### Tp[10] (Triphenyltin derivative of Schiff's base HL[10])

Yield (49%), m. p. 100°C, Elemental Analysis: calculated for $C_{32}H_{28}N_2OSn$: C, 66.81; H, 4.91; N, 4.87; found: C, 66.90; H, 4.85; N, 4.78; FT-IR (4000-400 $cm^{-1}$) 3020 ν(CH), 1645 ν(C=N), 1496 ν(CH=CH), 1174 ν(C–N), 1258ν(C–O), 454ν(Sn–N) $^1$H NMR ($DMSO_{4-D6}$, ppm), 7.54-7.56d; 7.24-7.27m; 7.2m (–$C_6H_5OH$), 6.84-6.87m; 7.07-7.09m; 6.91d (N–$C_6H_5$), 1.08s ($CH_3$–), 4.0s (–OH), 7.43m (Sn–$C_6H_5$), $^{13}$C NMR ($DMSO_4$–D6, ppm), 144.87 (C-1), 116.52 (C-2/5), 129.3 (C-3/4), 118.77 (C-6), 13.14 (C-7), 157.22 (C-8), 119.72 (C-9), 147.66 (C-10/14), 112.4 (C-11/13), 129.47 (C-12), 120.3 (C-15), 136.1 (C-16/20), 128.35 (C-17/19), 128.1 (C-18).

### Antibacterial activity

The antibacterial activity of triphenyltin derivatives of phenyl hydrazine Schiff's bases was tested against Escherichia coli and Staphylococcus aureus using the agar well diffusion method.[26] Cefexime and DMSO were used as standard drugs. The wells were dug in the media by using a sterile metallic borer with the centre at least 24 mm apart. The recommended concentration of the test sample (2 mg /ml in DMSO) was introduced into the respective wells. Other wells were supplemented with DMSO and reference antibacterial drugs serving as negative and positive controls, respectively. The plates were incubated immediately at 37°C for 20 h. The activity was determined by measuring the diameter of zones showing complete inhibition in millimetres. Growth inhibition was calculated with reference to positive control.

### Antifungal activity

The antifungal activity of synthesized triphenyltin derivatives of phenyl hydrazines Schiff's bases were tested against Aspergillus Flavus, Aspergillus Niger, Rhizoctonia Solani, Aspergillus Fumigatus and Mucorby using the tube diffusion test.[27] Terbinafine (200 mg/ ml) were used as standards drugs. Stock solutions of pure compounds (12 mg /ml) were prepared in sterile DMSO. Sabouraud dextrose agar was prepared by mixing sabouraud agar (32.5 g), glucose agar (4%) and agar-agar (4 g) in 500 ml of distilled water followed by steamed dissolution. Into screw-capped tubes, media (4 ml) was dispensed and autoclaved at 121°C for 15 min. Test compound (66.6 mg/ ml) was added from the stock solution to non-solidified Sabouraud agar media (50°C). The tubes were allowed to solidify at room temperature and were inoculated with 4 mm diameter portion of inoculums derived from a 7- day-old respective fungal culture. For non mycelial growth, an agar surface streak was employed. The tubes were incubated at 27-29°C for 7-10 days and the growth in the compound containing media was determined by measuring the linear growth (in mm) and growth inhibition and comparing it to the respective control. The amount of growth inhibition was calculated as:

Inhibition (%) = [(A-B)/B] × 100

A = Diameter of fungal colony in control plate

B = Diameter of fungal colony in test plate

## RESULTS AND DISCUSSION

The elemental analyses agree well with the proposed formula of the Schiff's bases and their metal complexes were prepared by a reaction of the Schiff's bases with the triphenyltin chloride in an anhydrous toluene medium all the triphenyltin derivatives of Phenylhydrazones were stable in air and soluble in most common organic

solvents. All Schiff's bases and there triphenyltin derivatives have sharp melting points and were characterized by FTIR and multinuclear NMR spectroscopic techniques.

IR spectra of Schiff's bases and their metal complexes were recorded in the range of 4000-400cm$^{-1}$. The complexation of tin with the ligand is confirmed by the absence of a broad band in the range of 400-500 cm$^{-1}$ due to ν (N-Sn).A single band at 3400-3300 cm$^{-1}$, characteristic for the NH group and present in the spectrum of the Schiff's bases but absent in triphenyltin derivatives of Schiff's bases.

In the $^{1}$H NMR spectra of all the Schiff's bases and their metal complexes have studied, All the protons present in the compounds have been identified by position and number.$^{13}$C NMR data of the studied compounds containing the carbons of the phenyl and alkyl groups attached to tin are observed and reported above. In-vitro antibacterial and antifungal activities of the synthesized Schiff's bases and there derivatives were carried out against pathogenic strains of bacteria and fungi.

**Table 1:** Antibacterial Activity of Schiff's Bases and there Triphenyltin Derivatives

| Triphenyltin derivatives of Schiff's bases | Zone of inhibition(mm) | |
|---|---|---|
| | Staph. Aureus | E. Coli |
| HL$^{6}$ | 8 | 6 |
| Tp$^{6}$ | 0 | 0 |
| HL$^{7}$ | 0 | 11 |
| Tp$^{7}$ | 0 | 0 |
| HL$^{8}$ | 0 | 6 |
| Tp$^{8}$ | 15 | 0 |
| HL$^{9}$ | 7 | 10 |
| Tp$^{9}$ | 0 | 0 |
| HL$^{10}$ | 0 | 7 |
| Tp$^{10}$ | 13 | 0 |
| Cefexime | 21 | 21 |
| DMSO | 0 | 0 |

**Table 2:** Antifungal Activity of Schiff's Bases and there Triphenyltin Derivatives

| Triphenyltin derivatives of Schiff's bases | Zone of inhibition | | | | |
|---|---|---|---|---|---|
| | Aspergillus Flavus | Aspergillus Niger | Rhizoctonia Solani | Aspergillus Fumigatus | Mucor |
| HL$^{6}$ | 6 | 0 | 9 | 0 | 0 |
| Tp$^{6}$ | 8 | 11 | 0 | 6 | 0 |
| HL$^{7}$ | 0 | 0 | 0 | 11 | 15 |
| Tp$^{7}$ | 10 | 17 | 0 | 16 | 16 |
| HL$^{8}$ | 9 | 7 | 10 | 0 | 13 |
| Tp$^{8}$ | 0 | 0 | 0 | 6 | 9 |
| HL$^{9}$ | 0 | 0 | 0 | 0 | 0 |
| Tp$^{9}$ | 11 | 8 | 0 | 9 | 13 |
| HL$^{10}$ | 0 | 0 | 0 | 0 | 18 |
| Tp$^{10}$ | 9 | 7 | 0 | 8 | 11 |
| Terbinafine | 28 | 33 | 35 | 30 | 33 |

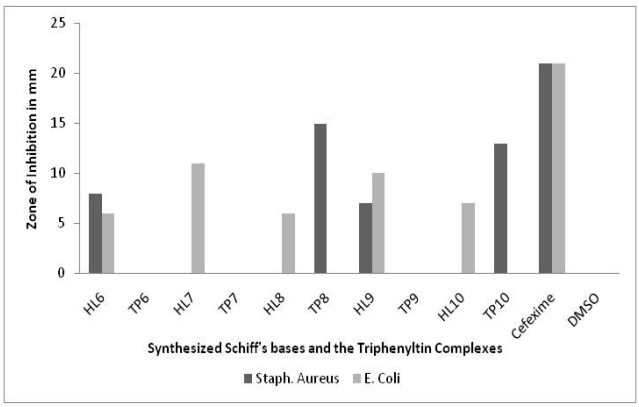


**Figure 3:** Graphical Description of the Antibacterial Assay

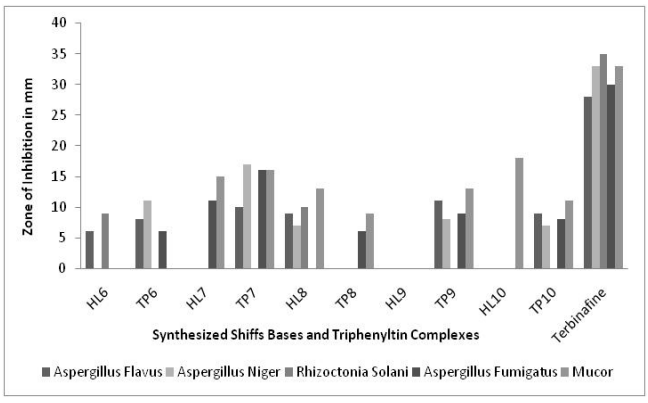


**Figure 4:** Graphical Description of the Antifungal Assay

The results are given below in table 1 and table 2 with their graphical illustrations in figure 3 and figure 4 respectively.

## CONCLUSION

Physical and chemical characterization justifies the synthesis of the Schiff's bases and their triphenyltin

derivatives. The results revealed that some of the compounds possess antibacterial and antifungal properties. However, further chemical work and biological investigations are required to identify the other possible therapeutic actions of the synthesized compounds. This study can also be fruitful for the development of other novel metal complexes of the synthesized Schiff's bases as interesting biologically active molecules for future evaluation.

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