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# Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry

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# Synthesis and Characterization of Biologically Active Organosilicon(IV) Complexes with Schiff Bases Derived from o-aminothiophenol

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The synthesis and structural features of some organosilicon(IV) complexes with Schiff bases derived by condensing Substituted benzaldehyde with o-aminothiophenol have been discussed. The newly synthesized compounds have been characterized by elemental analyses, IR, <sup>1</sup>H, <sup>13</sup>C, and <sup>29</sup>Si NMR spectral studies. With the help of spectral studies, penta, and hexacoordinated environments around the central silicon atoms in the 1:1 and 1:2 complexes, respectively, have been proposed. Some have been tested for the growth inhibitory activity against some bacterial and yeast strains *in vitro*. The antimicrobial studies revealed that the Schiff bases and some of their metal complexes possess more healing activity.

Keywords antimicrobial activity, o-aminothiophenol, organosilicon(IV), Schiff bases, spectral studies

#### Abbreviations

DMSO-Dimethylsulphoxide, IR-Infra red, NMR-Nuclear Magnetic Resonance, MIC-Minimum Inhibitory Concentration, MBC- Minimum Bactericidal Concentration, MHB-Mullen Hinton Broth, MHA- Mullen Hinton Agar.

# INTRODUCTION

Interaction of metal ions with N, O, and S containing organic moieties has attracted much attention in recent years.<sup>[1,2]</sup> Schiff bases and their metal complexes have exhibited biological activity as antibiotics, antiviral, and antitumor agents because of their specific structures. Hetronuclear Schiff base complexes have been found in applications as magnetic materials, catalysts, and in the biological engineering field.<sup>[3–6]</sup> In addition,

the presence of both a hard and a soft donor group in one ligand increases the coordination ability towards hard as well as soft acidic metals. Organosilicon compounds of sulphur containing ligands have attracted much attention recently due to their biological importance. The sulphur containing ligands are well known for their anticarcinogenic, antibacterial, tuberoculostic, antifungal, insecticidal, and acarcidal activities. It has been reported that the activity of sulphur containing ligand increases on complexation.<sup>[7-13]</sup> The interest in organosilicon(IV) compounds is generated due to their versatile applicability in pharmaceutical and in chemical industries. Generally organosilicon compounds seem to own their antitumour properties to the immunodefensive system of organism.<sup>[14-17]</sup>The medical application and effectiveness of the silatranes in the treatment of wounds and tumors are thought to be related to the role of silicon in the growth of epithelial and connective tissue and hair, where its function is to impart strengths, elasticity, and impermeability to water.<sup>[18]</sup> Prompted from these application of organosilicon compounds, we reported the reaction of monofunctional bidentate Schiff bases with organosilicon(IV)halide, characterization of newly prepared complexes, and the antimicrobial activity of the complexes so formed. The Schiff bases were prepared by condensing different substituted benzaldehyde with o-aminothiophenol. The structure and abbreviation of Schiff bases are given in Figure 1.

#### **EXPERIMENTAL**

#### Reagents

Due to the moisture sensitive nature of the starting materials and reaction products, strict precaution were taken to exclude moisture by using guard tube packed with anhydrous calcium chloride and dried glass apparatus. The glass fitted with interchangeable standards ground joints was used throughout these investigations. All chemicals used were of analytical reagent grade and of highest purity available. All the organic solvents used during experiment were dried and purified by standard methods.

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FIG. 1. Structure of ligands, where  $HL^1$ : 2-Chloro-5-nitrobenzylideneo-aminothiophenol;  $HL^2$ : 3-Bromobenzylidene-o-aminothiophenol;  $HL^3$ : 4-Methylbenzylidene-o-aminothiophenol; and  $HL^4$ : 2-Chlorobenzylidene-oaminothiophenol.

#### Physical Measurements and Analytical Methods

Carbon, hydrogen and nitrogen were estimated by semi micro analyzer, LG-VEG Labogerate and orthopedic Leipzig (SAIF, Punjab University Chandigarh, India). Sulphur and Chlorine were estimated by the *Messenger's* and *Volhard's* methods,<sup>[29]</sup> respectively. Silicon was determined gravimetrically as SiO<sub>2</sub>. Molecular weights were determined by the Rast Camphor method (freezing point depression method) using resublimed camphor (m.pt. 178°C).

I.R. spectra were recorded in KBr pellets using Perkin-Elmer 842 spectrophotometer in 4000–450 cm<sup>-1</sup>region. <sup>1</sup>H, <sup>13</sup>C NMR spectra were recorded in DMSO-d<sub>6</sub> on a Bruker Avance II 400 NMR spectrometer at 400 MHz with reference to Me<sub>4</sub>Si (0.0 ppm). Melting points were determined in sealed capillary in electrical melting point apparatus.

#### Preparation of the Ligands (HL<sup>1</sup>-HL<sup>4</sup>)

The ligands  $HL^{1,2,4}$  were prepared by condensing o-aminothiophenol with 2-chloro-5-nitrobenzaldehyde, 3bromobenzaldehyde, and 2-chlorobenzaldehyde in equimolar ratio in absolute ethanol. The contents were refluxed for 4–5 hours, and solid was obtained after keeping overnight, washed with ethanol, and dried *in vacuo* after recrystalization from same solvent. The ligand  $HL^3$  was prepared by condensing o-aminothiophenol with 4-methylbenzaldehyde in dry ethanol under ice cold condition. The physical properties and microanalysis of these are recorded in Table 1.

#### Synthesis of the Organosilicon(IV) Complexes

For the synthesis of complexes first sodium salts of Schiff base were prepared by reacting equimolar ratio of ligand and sodium metal in dried methanol (20 mL). To a weighed amount of diorganosilicon(IV)dichloride in dry methanol was added the corresponding amount of the sodium salt of the ligands in 1:1 and 1:2 molar ratios. The reaction mixtures were refluxed for a period of 12–13 hrs. The white precipitate of sodium chloride formed during the course of the reaction was removed by filtration and the filtrate was dried under reduced pressure. The re-

$$Me_{2}SiCl_{2} + N S Na \xrightarrow{MeOH} Me_{2}Si(N S)Cl + NaCl$$

$$Me_{2}SiCl_{2} + 2N S Na \xrightarrow{MeOH} Me_{2}Si(NS)_{2} + 2 NaCl$$

$$SCH. 1.$$

sulting products were repeatedly washed with different dried organic solvents and finally dried *in vacuo* for 1–2 hrs. The details of the analyses of the resulting products are recorded in Table 1.

#### **RESULTS AND DISCUSSION**

The reaction of dimethylsilicondichloride with mono functional bidentate Schiff bases (HL<sup>1</sup>-HL<sup>4</sup>) in 1:1 and 1:2 molar ratios in dry methanol proceed as shown in Scheme 1.

All the newly synthesized complexes are colored solid, and soluble in most of the common organic solvents. These have been found to be monomeric as evident by their molecular weight determination. The physical properties and microanalysis of ligands and silicon complexes are recorded in Table 1.

#### I.R. Spectra and Mode of Bonding

IR spectral data of the ligands and their metal complexes have been interpreted on the basis of available literature data. The IR data of the Schiff base ligands and their complexes are presented in Table 2. The IR spectra of the complexes were compared with those of the free ligands in order to determine the coordination sites that may be involved in chelation. There are some guide peaks in the spectra of the ligands, which are helpful in achieving this goal. The position and/or the intensities of these peaks are expected to change upon chelation.

IR spectra of the ligands, show a sharp band at 1605  $\pm$ 5 cm<sup>-1</sup> due to  $\nu$ (>C=N) frequency of the free azomethine group shifts to the lower frequency ( $\sim 20 \text{ cm}^{-1}$ ) in the silicon complexes indicating the coordination of the azomethine nitrogen to the silicon atom.<sup>[1,7,19]</sup> A shift of this frequency to the higher side, as well as lower wave number side, has been reported in the literature.<sup>[14]</sup> The ligands showed a weak absorption band in the region 2300–2400 cm<sup>-1</sup> due to  $\nu$ (S-H).<sup>[1]</sup> This band due to v(S-H) completely disappeared in the case of metal complexes indicating deprotonation of thiophenolic protons prior to coordination. The coordination of sulphur atom to silicon atom also ascertained from v asym and sym(C-S) peaks at  $\sim 693$  cm<sup>-1</sup> and  $\sim 732$  cm<sup>-1</sup>, respectively, are observed in the free ligand and shifted to lower frequency in the spectra of the metal complexes.<sup>[20]</sup> In the spectra of metal complexes bands due to asymmetric and symmetric deformation of  $\nu$ (CH<sub>3</sub>-Si) group were observed at ~ 1420cm<sup>-1</sup> and 1271cm<sup>-1</sup>, respectively.<sup>[7]</sup> The new bands were observed in the spectra of the silicon complexes at 572–586  $\text{cm}^{-1}$  and 550–566  $\text{cm}^{-1}$ due to  $\nu$ (Si-N) and  $\nu$ (Si-S) vibrations, respectively,<sup>[21]</sup> which were absent in the spectra of the free ligands supporting the coordination through sulphur and azomethine nitrogen atoms. However, no large change in the symmetric and asymmetric

				Ш	lemental ana	ılysis (%)				
ompound	Color and state	Yield (%)	M P (°C)	C Found (calcd.)	H Found (calcd.)	N Found (calcd.)	S Found (calcd.)	Si Found (calcd.)	Mol. wt. Found (calcd.)	
$L^{1}$ (C <sub>13</sub> H <sub>9</sub> N <sub>2</sub> O <sub>2</sub> SCI)	Reddish solid	76	123-125	52.91 (53.34)	3.10 (3.09)	9.18 (9.56)	10.29 (10.95)	I	278.31 (292.73)	
$L^{2}$ (C <sub>13</sub> H <sub>10</sub> NSBr)	Brown solid	74	86-88	52.83 (53.44)	3.32 (3.44)	4.67 (4.79)	10.24 (10.97)	Ι	281.73 (292.17)	
$L^{3}$ (C <sub>14</sub> H <sub>13</sub> NS)	Greenish yellow solid	80	67–68	73.69 (73.97)	5.14 (5.76)	5.82 (6.15)	13.82 (14.10)	I	221.13 (227.31)	
$L^{4}$ (C <sub>13</sub> H <sub>10</sub> NSCI)	Brown solid	69	70–72	62.76 (63.02)	3.89 (4.06)	5.53 (5.65)	12.13 (12.94)	I	236.32 (247.73)	
$le_2Si(L^1)Cl$	Cream solid	73	142–145	45.43 (46.75)	3.59 (3.66)	7.16 (7.26)	8.28 (8.32)	6.91 (7.28)	364.67 (385.30)	
$(SiC_{15}H_{14}N_2O_2SCl_2)$										
$fe_2Si(L^1)_2$	Orange yellow	74	165-170	51.89 (52.42)	3.31 (3.45)	8.63 (8.72)	9.87 (9.99)	4.18 (4.37)	603.21 (641.55)	
$(SiC_{28}H_{22}N_4O_4S_2Cl_2)$	) solid									
$le_2Si(L^2)Cl$	Violet solid	71	205-210	46.13 (46.82)	3.90 (3.92)	3.51 (3.63)	8.31 (8.33)	7.21 (7.29)	351.23 (384.77)	
(SiC <sub>15</sub> H <sub>15</sub> NSBrCl)										
$1e_2Si (L^2)_2$	Bluish green	78	217-220	52.12 (52.50)	3.59 (3.77)	4.27 (4.37)	9.93(10.01)	4.28 (4.38)	612.67 (640.49)	
$(SiC_{28}H_{24}N_2S_2Br_2)$	solid									
$le_2Si(L^3)Cl$	Green solid	68	160-165	59.43 (60.07)	5.58 (5.66)	4.27 (4.37)	9.88 (10.02)	8.46 (8.77)	301.98 (319.91)	
(SiC <sub>16</sub> H <sub>18</sub> NSCI)										
$le_2Si(L^3)_2$	Yellowish brown	<i>6L</i>	175-180	70.29 (70.54)	5.78 (5.91)	5.47 (5.49)	12.48 (12.55)	5.35 (5.49)	478.77 (510.76)	
$(SiC_{30}H_{30}N_2S_2)$	solid									
$le_2Si(L^4)Cl$	Grayish solid	81	120-123	52.84 (52.93)	4.35 (4.43)	3.89 (4.11)	9.34 (9.42)	8.19 (8.25)	318.13 (340.33)	
(SiC <sub>15</sub> H <sub>15</sub> NSCl <sub>2</sub> )										
$1e_2Si(L^4)_2$	Yellowish green	82	166–168	60.83 (60.96)	4.27 (4.38)	4.83 (5.07)	11.43 (11.62)	4.84 (5.09)	512.78 (551.61)	
	$\begin{array}{c} {\rm L}^1 ({\rm C}_{13}{\rm H}_{10}{\rm NSBr}) \\ {\rm L}^2 ({\rm C}_{13}{\rm H}_{10}{\rm NSBr}) \\ {\rm L}^2 ({\rm C}_{13}{\rm H}_{10}{\rm NSBr}) \\ {\rm L}^2 ({\rm C}_{13}{\rm H}_{10}{\rm NSBr}) \\ {\rm L}^4 ({\rm C}_{13}{\rm H}_{10}{\rm NSCl}) \\ {\rm fe}_2 {\rm Si}({\rm L}^1) {\rm Cl} \\ ({\rm SiC}_{15}{\rm H}_{14}{\rm N}_2{\rm O}_2 {\rm SCl}_2) \\ {\rm fe}_2 {\rm Si}({\rm L}^1)_2 \\ ({\rm SiC}_{15}{\rm H}_{14}{\rm N}_2{\rm O}_2 {\rm SCl}_2) \\ {\rm fe}_2 {\rm Si}({\rm L}^2)_2 \\ ({\rm SiC}_{15}{\rm H}_{15}{\rm NSBrCl}) \\ {\rm fe}_2 {\rm Si}({\rm L}^2)_2 \\ ({\rm SiC}_{15}{\rm H}_{15}{\rm NSBrCl}) \\ {\rm fe}_2 {\rm Si}({\rm L}^3)_2 \\ ({\rm SiC}_{16}{\rm H}_{18}{\rm NSCl}) \\ {\rm fe}_2 {\rm Si}({\rm L}^3)_2 \\ ({\rm SiC}_{10}{\rm H}_{30}{\rm N}_2 {\rm S}_2) \\ {\rm fe}_2 {\rm Si}({\rm L}^4)_2 \end{array} \end{array}$	L1(C13H_0N2O_2SCI)Reddish solidL2(C13H_10NSBr)Brown solidL3(C14H_{13}NS)SolidL4(C13H_10NSCI)Brown solidGreenish yellowsolid $(SiC_{15}H_{14}N_2O_2SCI_2)$ Brown solid $(SiC_{15}H_{14}N_2O_2SCI_2)$ Brown solid $(SiC_{15}H_{14}N_2O_2SCI_2)$ Brown solid $(SiC_{15}H_{14}N_2O_2SCI_2)$ Orange yellow $(SiC_{15}H_{15}NSBrCI)$ Orange yellow $(SiC_{28}H_{22}N_4O_4S_2CI_2)$ Solid $fe_2Si(L^2)CI$ Violet solid $(SiC_{28}H_{24}N_2S_2Br_2)$ Solid $fe_2Si(L^3)CI$ Green solid $(SiC_{30}H_{30}N_2S_2)$ Solid $fe_2Si(L^4)CI$ Grayish brown $(SiC_{15}H_{15}NSCI_2)$ Yellowish brown $(SiC_{15}H_{15}NSCI_2)$ Yellowish brown $(SiC_{15}H_{15}NSCI_2)$ Yellowish brown $(SiC_{15}H_{15}NSCI_2)$ Yellowish brown	L1Clash solid76L2(Cl3H10NSBr)Brown solid76L2(Cl3H10NSBr)Brown solid74L3(Cl4H13NS)Greenish yellow80solidBrown solid73 $(SiC_{15}H_{14}N_2O_2SCl_2)$ Brown solid73 $(SiC_{15}H_{14}N_2O_2SCl_2)$ Brown solid73 $(SiC_{15}H_{14}N_2O_2SCl_2)$ Orange yellow74 $(SiC_{15}H_{14}N_2O_2SCl_2)$ Orange yellow74 $(SiC_{28}H_{24}N_2S_2Br_2)$ Orange yellow74 $(SiC_{28}H_{24}N_2S_2Br_2)$ solid71 $(SiC_{28}H_{24}N_2S_2Br_2)$ Solid78 $(SiC_{28}H_{24}N_2S_2Br_2)$ solid68 $(SiC_{28}H_{24}N_2S_2)$ Solid68 $(SiC_{26}H_{18}NSCl)$ solid79 $(SiC_{20}H_{30}N_2S_2)$ solid79 $(SiC_{23}H_{15}NSCl_2)$ Solid81 $(SiC_{21}H_{15}NSCl_2)$ Solid81 $(SiC_{21}H_{15}NSCl_2)$ Solid81 $(SiC_{21}H_{15}NSCl_2)$ Solid81 $(SiC_{21}H_{15}NSCl_2)$ Solid81 $(SiC_{21}H_{15}NSCl_2)$ Solid81	L <sup>1</sup> (C <sub>13</sub> H <sub>0</sub> N <sub>2</sub> O <sub>2</sub> SCI)         Reddish solid         76         123-125           L <sup>2</sup> (C <sub>13</sub> H <sub>10</sub> NSBr)         Brown solid         76         123-125           L <sup>3</sup> (C <sub>14</sub> H <sub>13</sub> NS)         Brown solid         76         123-125           L <sup>4</sup> (C <sub>13</sub> H <sub>10</sub> NSBr)         Brown solid         76         123-125           L <sup>4</sup> (C <sub>13</sub> H <sub>10</sub> NSCI)         Brown solid         79         67-68           Solid         Cream solid         73         142-145           (SiC <sub>15</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> SCI <sub>2</sub> )         Brown solid         69         70-72           (SiC <sub>15</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> SCI <sub>2</sub> )         Brown solid         73         142-145           (SiC <sub>15</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> SCI <sub>2</sub> )         Cream solid         74         165-170           (SiC <sub>28</sub> H <sub>22</sub> N <sub>4</sub> O <sub>4</sub> S <sub>2</sub> CI <sub>2</sub> )         Solid         71         205-210           (SiC <sub>28</sub> H <sub>22</sub> N <sub>4</sub> O <sub>4</sub> S <sub>2</sub> CI <sub>2</sub> )         Solid         71         205-210           (SiC <sub>28</sub> H <sub>22</sub> N <sub>16</sub> NSBrCI)         Violet solid         71         205-210           (SiC <sub>16</sub> H <sub>18</sub> NSCI)         Violet solid         71         205-210           (SiC <sub>28</sub> H <sub>24</sub> N <sub>2</sub> S <sub>2</sub> Br <sub>2</sub> )         Solid         68         160-165           (SiC <sub>18</sub> H <sub>15</sub> NSCI)         Solid         68         160-165           (SiC <sub>28</sub> H <sub>24</sub> N <sub>2</sub> S <sub>2</sub> Br <sub>2</sub> )	L <sup>1</sup> (C <sub>13</sub> H <sub>10</sub> NSBr)         Constant of the solid         76         123-125         52.91 (53.34)           L <sup>2</sup> (C <sub>13</sub> H <sub>10</sub> NSBr)         Brown solid         74         86–88         52.83 (53.44)           L <sup>3</sup> (C <sub>14</sub> H <sub>13</sub> NS)         Brown solid         74         86–88         52.91 (53.34)           L <sup>3</sup> (C <sub>14</sub> H <sub>13</sub> NS)         Brown solid         74         86–88         52.83 (53.44)           L <sup>4</sup> (C <sub>13</sub> H <sub>10</sub> NSC1)         Brown solid         74         86–88         52.83 (53.24)           Redish value         69         70–72         62.76 (63.02)         801d           SiC <sub>15</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> SC1 <sub>2</sub> Brown solid         73         142–145         45.43 (46.75)           (SiC <sub>15</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> SC1 <sub>2</sub> )         Cream solid         74         165–170         51.89 (52.42)           (SiC <sub>15</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> SC1 <sub>2</sub> )         Solid         71         205–210         46.13 (46.82)           (SiC <sub>15</sub> H <sub>14</sub> N <sub>2</sub> O <sub>2</sub> SC1 <sub>2</sub> )         solid         71         205–210         51.89 (52.42)           (SiC <sub>15</sub> H <sub>15</sub> NSBrC1)         solid         71         205–210         46.13 (46.82)           (SiC <sub>15</sub> H <sub>15</sub> NSBrC1)         solid         71         205–210         46.13 (46.82)           (SiC <sub>15</sub> H <sub>15</sub> NSBrC1)         solid         71         205–2	L <sup>1</sup> (C <sub>13</sub> H <sub>9</sub> N <sub>2</sub> O <sub>2</sub> SCI)         Reddish solid         76         123-125         52.91         53.44         3.32         3.44           L <sup>3</sup> (C <sub>14</sub> H <sub>13</sub> NS)         Brown solid         74         86–88         52.83         53.44         3.32         3.44           L <sup>3</sup> (C <sub>14</sub> H <sub>13</sub> NS)         Greenish yellow         80         67–68         73.69         (73.97)         5.14         5.76)           L <sup>3</sup> (C <sub>14</sub> H <sub>13</sub> NS)         Greenish yellow         80         67–68         73.69         (73.97)         5.14         5.76)           Redish solid         74         86–88         52.83         (53.44)         3.32         (3.45)           Reside         73         97–72         62.76         (63.02)         3.89         (4.06)           Reside         73         142–145         45.43         (46.75)         3.59         (3.66)           Reside         73         142–145         45.43         (46.75)         3.30         (3.92)           Reside         73         142–145         45.43         (46.75)         3.59         (3.66)           Reside         73         142–145         45.43         (46.75)         3.30         (3.92)           Reside         71 <td>L<sup>1</sup> (C<sub>13</sub>H<sub>9</sub>N<sub>2</sub>O<sub>2</sub>SCI)         Reddish solid         76         123-125         52.91         53.3.41         3.10         3.09         9.18         9.56           L<sup>2</sup> (C<sub>13</sub>H<sub>10</sub>NSBr)         Brown solid         74         86–88         52.83         53.44         3.56         5.82         6.15           L<sup>3</sup> (C<sub>14</sub>H<sub>13</sub>NS)         Greenish yellow         80         67–68         73.69         73.97         5.14         5.76         5.82         6.15           L<sup>4</sup> (C<sub>13</sub>H<sub>10</sub>NSCI)         Brown solid         74         86–88         52.83         53.44         3.70         5.14         5.76         5.82         6.15           R<sup>2</sup> (C<sub>13</sub>H<sub>10</sub>NSCI)         Brown solid         74         86–88         52.43         46.75         3.29         4.06         5.53         6.5</td> <td>L<sup>1</sup> (C<sub>13</sub>H<sub>9</sub>N<sub>2</sub>O<sub>2</sub>SCI)         Reddish solid         76         123-125         52.91 (53.34)         3.10 (3.09)         9.18 (9.56)         10.29 (10.95)           L<sup>2</sup> (C<sub>13</sub>H<sub>10</sub>NSBr)         Brown solid         76         82.83 (53.34)         3.30 (3.09)         9.18 (9.56)         10.29 (10.97)           L<sup>4</sup> (C<sub>13</sub>H<sub>10</sub>NS)         Greenish yellow         80         67-68         73.69 (73.97)         5.14 (5.76)         5.82 (6.15)         13.82 (14.10)           R<sup>4</sup> (C<sub>13</sub>H<sub>10</sub>NSCI)         Brown solid         69         70-72         62.76 (63.02)         3.89 (4.06)         5.53 (5.65)         12.13 (12.94)           R<sup>2</sup> (C<sub>13</sub>H<sub>10</sub>NSCI)         Brown solid         69         70-72         62.76 (63.02)         3.89 (4.06)         5.53 (5.65)         12.13 (12.94)           R<sup>2</sup> Si(L<sup>1</sup>)<sub>C</sub>         Cream solid         73         142-145         45.43 (46.75)         3.59 (3.66)         7.16 (7.26)         8.28 (8.32)           R<sup>2</sup> Si(L<sup>1</sup>)<sub>L</sub>         Orange yellow         74         165-170         51.89 (52.42)         3.31 (3.45)         8.31 (8.33)           R<sup>2</sup> Si(L<sup>1</sup>)<sub>L</sub>         Orange yellow         71         253 (5.65)         3.21 (3.63)         8.31 (8.33)           R<sup>2</sup> Si(L<sup>1</sup>)<sub>L</sub>         Orange yellow         71         165 (5.66)         4.27 (4.37)</td> <td>L<sup>1</sup> (C<sub>13</sub>H<sub>0</sub>NSBr)         Brown solid         76         123-125         52.91 (53.34)         3.10 (3.09)         9.18 (9.56)         10.29 (10.95)         -           L<sup>2</sup> (C<sub>13</sub>H<sub>0</sub>NSBr)         Brown solid         74         86-88         52.83 (53.44)         3.32 (3.44)         4.67 (4.79)         10.24 (10.97)         -           L<sup>3</sup> (C<sub>13</sub>H<sub>10</sub>NSCl)         Brown solid         76         123-125         52.91 (53.02)         3.89 (4.06)         5.53 (6.15)         13.82 (14.10)         -           L<sup>4</sup> (C<sub>13</sub>H<sub>10</sub>NSCl)         Brown solid         69         70-72         62.76 (63.02)         3.89 (4.06)         5.53 (5.55)         12.13 (12.94)         -           Re<sup>5</sup>Si(L<sup>3</sup>)Cl         Brown solid         69         70-72         62.76 (63.02)         3.80 (4.06)         5.53 (5.57)         13.82 (14.10)         -           Re<sup>5</sup>Si(L<sup>3</sup>)Cl         Brown solid         69         70-72         62.76 (63.02)         3.80 (4.06)         5.33 (8.72)         9.81 (7.28)           SiC(2<sub>3</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>SCl<sup>2</sup>)         Orange yellow         74         165-170         51.89 (52.42)         3.31 (3.45)         8.63 (8.72)         9.87 (9.99)         4.18 (4.37)           SiC(2<sub>3</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>SCl         Violet solid         71         125-12 (52.25)         3.31 (3.46.82)</td> <td>L<sup>1</sup> (C<sub>13</sub>H<sub>10</sub>NSDr)         Control (1, 1, 1, 0)         Control (1, 2, 2, 2)         Control (2, 2)         <thcontrol (2,="" 2)<="" th="">         Control (2, 2)</thcontrol></td>	L <sup>1</sup> (C <sub>13</sub> H <sub>9</sub> N <sub>2</sub> O <sub>2</sub> SCI)         Reddish solid         76         123-125         52.91         53.3.41         3.10         3.09         9.18         9.56           L <sup>2</sup> (C <sub>13</sub> H <sub>10</sub> NSBr)         Brown solid         74         86–88         52.83         53.44         3.56         5.82         6.15           L <sup>3</sup> (C <sub>14</sub> H <sub>13</sub> NS)         Greenish yellow         80         67–68         73.69         73.97         5.14         5.76         5.82         6.15           L <sup>4</sup> (C <sub>13</sub> H <sub>10</sub> NSCI)         Brown solid         74         86–88         52.83         53.44         3.70         5.14         5.76         5.82         6.15           R <sup>2</sup> (C <sub>13</sub> H <sub>10</sub> NSCI)         Brown solid         74         86–88         52.43         46.75         3.29         4.06         5.53         6.5	L <sup>1</sup> (C <sub>13</sub> H <sub>9</sub> N <sub>2</sub> O <sub>2</sub> SCI)         Reddish solid         76         123-125         52.91 (53.34)         3.10 (3.09)         9.18 (9.56)         10.29 (10.95)           L <sup>2</sup> (C <sub>13</sub> H <sub>10</sub> NSBr)         Brown solid         76         82.83 (53.34)         3.30 (3.09)         9.18 (9.56)         10.29 (10.97)           L <sup>4</sup> (C <sub>13</sub> H <sub>10</sub> NS)         Greenish yellow         80         67-68         73.69 (73.97)         5.14 (5.76)         5.82 (6.15)         13.82 (14.10)           R <sup>4</sup> (C <sub>13</sub> H <sub>10</sub> NSCI)         Brown solid         69         70-72         62.76 (63.02)         3.89 (4.06)         5.53 (5.65)         12.13 (12.94)           R <sup>2</sup> (C <sub>13</sub> H <sub>10</sub> NSCI)         Brown solid         69         70-72         62.76 (63.02)         3.89 (4.06)         5.53 (5.65)         12.13 (12.94)           R <sup>2</sup> Si(L <sup>1</sup> ) <sub>C</sub> Cream solid         73         142-145         45.43 (46.75)         3.59 (3.66)         7.16 (7.26)         8.28 (8.32)           R <sup>2</sup> Si(L <sup>1</sup> ) <sub>L</sub> Orange yellow         74         165-170         51.89 (52.42)         3.31 (3.45)         8.31 (8.33)           R <sup>2</sup> Si(L <sup>1</sup> ) <sub>L</sub> Orange yellow         71         253 (5.65)         3.21 (3.63)         8.31 (8.33)           R <sup>2</sup> Si(L <sup>1</sup> ) <sub>L</sub> Orange yellow         71         165 (5.66)         4.27 (4.37)	L <sup>1</sup> (C <sub>13</sub> H <sub>0</sub> NSBr)         Brown solid         76         123-125         52.91 (53.34)         3.10 (3.09)         9.18 (9.56)         10.29 (10.95)         -           L <sup>2</sup> (C <sub>13</sub> H <sub>0</sub> NSBr)         Brown solid         74         86-88         52.83 (53.44)         3.32 (3.44)         4.67 (4.79)         10.24 (10.97)         -           L <sup>3</sup> (C <sub>13</sub> H <sub>10</sub> NSCl)         Brown solid         76         123-125         52.91 (53.02)         3.89 (4.06)         5.53 (6.15)         13.82 (14.10)         -           L <sup>4</sup> (C <sub>13</sub> H <sub>10</sub> NSCl)         Brown solid         69         70-72         62.76 (63.02)         3.89 (4.06)         5.53 (5.55)         12.13 (12.94)         -           Re <sup>5</sup> Si(L <sup>3</sup> )Cl         Brown solid         69         70-72         62.76 (63.02)         3.80 (4.06)         5.53 (5.57)         13.82 (14.10)         -           Re <sup>5</sup> Si(L <sup>3</sup> )Cl         Brown solid         69         70-72         62.76 (63.02)         3.80 (4.06)         5.33 (8.72)         9.81 (7.28)           SiC(2 <sub>3</sub> H <sub>12</sub> N <sub>2</sub> O <sub>2</sub> SCl <sup>2</sup> )         Orange yellow         74         165-170         51.89 (52.42)         3.31 (3.45)         8.63 (8.72)         9.87 (9.99)         4.18 (4.37)           SiC(2 <sub>3</sub> H <sub>12</sub> N <sub>2</sub> O <sub>2</sub> SCl         Violet solid         71         125-12 (52.25)         3.31 (3.46.82)	L <sup>1</sup> (C <sub>13</sub> H <sub>10</sub> NSDr)         Control (1, 1, 1, 0)         Control (1, 2, 2, 2)         Control (2, 2) <thcontrol (2,="" 2)<="" th="">         Control (2, 2)</thcontrol>

Yellowish green solid

 $(SiC_{28}H_{24}N_2S_2Cl_2)$ 

Analysis and physical properties of the ligands and their silicon complexes TABLE 1

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Sr. no.	Compound	$\nu(C=N)$	ν( <b>S-H</b> )	$\nu$ (C-S) sym asym	v(Si-N)	v(Si-S)	ν(CH <sub>3</sub> -Si) asym sym	$\nu(NO_2)$ sym asym
1.	$HL^1$	1604	2390	737	_	_	_	1348
				695				1516
2.	$HL^4$	1607	2366	731	-	_	_	_
				692				
3.	Me <sub>2</sub> Si(L <sup>1</sup> )Cl	1588	_	731	586	566	1420	1344
				694			1273	1523
4.	$Me_2Si(L^1)_2$	1596	_	729	586	565	1423	1345
				694			1277	1522
5.	Me <sub>2</sub> Si(L <sup>4</sup> )Cl	1585	_	731	573	552	1428	—
				685			1271	
6.	$Me_2Si(L^4)_2$	1590	_	730	572	550	1426	—
				684			1270	

TABLE 2 I.R. spectral data  $(cm^{-1})$  of the ligands  $[HL^1, HL^4]$  and their metal complexes

modes of NO<sub>2</sub> group, which appeared at ~ 1345 cm<sup>-1</sup> and 1520 cm<sup>-1</sup>, respectively<sup>[22]</sup> in one of the ligand [HL<sup>1</sup>]. The presence of only one  $\nu$ (Si-N) band<sup>[14]</sup> in metal complexes suggests that the 1:2 metal complexes exist in the trans form.

### <sup>1</sup>H NMR Spectra

The proton magnetic resonance spectral data of ligands and its corresponding silicon complexes have been recorded in DMSO-d<sub>6</sub>. The chemical shift values relative to the TMS peak are listed in Table 3.The broad signal present at  $\delta$  3.61– 4.31 ppm in the free ligands<sup>[23]</sup> is due to –SH protons, disappeared in the spectra of their 1:1 and 1:2 complexes, thereby indicating the deprotonation of the functional group and complexation with the silicon atom. In fact the aromatic regions of the spectra are complicated due the overlapping of several signals that have precluded the identification of individual resonances. However, the direct comparison of the intensity of the

aromatic region protons signals with that of the clearly observable azomethine proton (-HC=N-) in the downfield region and methyl protons for the complexes reveals the presence of the expected number of protons for all the complexes. The signal at  $\delta$  8.21–8.59 ppm in the free ligands due to azomethine protons are shifted <sup>[1,19,23]</sup> in the spectra of their 1:1 and 1:2 metal complexes (Table 3). This indicates the chelation of the ligands through azomethine nitrogen with silicon atom. The methyl protons (Si-CH<sub>3</sub>) are observed in the region,  $\delta$  1.61–1.20 ppm in the spectra of the complexes.

# <sup>13</sup>C NMR Spectra

The <sup>13</sup>C NMR spectra of Schiff bases 2-Chloro-5-nitro benzylidene-o-aminothiophenol [HL<sup>1</sup>], 4-Methylbenzylidene-o-aminothiophenol [HL<sup>3</sup>] and their corresponding 1:1 and 1:2 metal complexes have been interpreted on the basis of available literature data (Table 4).<sup>[21,24]</sup> The chemical shift values of the

TABLE 3	
<sup>1</sup> H NMR spectral data ( $\delta$ , ppm) of ligands and metal comp	olexes

Sr. no.	Compounds	-HC=N-	—SH	Aromatic –H	Si–CH <sub>3</sub>	Aromatic – CH <sub>3</sub>
1.	$HL^1$	8.59(s, 1H)	4.31(s, 1H)	8.11–6.84(m, 7H)	_	_
2.	$Me_2Si(L^1)Cl$	8.72(s, 1H)	_	8.56–6.86(m, 7H)	1.61(s, 3H)	_
3.	$Me_2Si(L^1)_2$	8.81(s, 1H)	_	8.61-6.85(m, 7H)	1.42(s, 3H)	_
4.	$HL^2$	8.21(s, 1H)	3.61(s, 1H)	7.25-6.55(m, 8H)	-	_
5.	$Me_2Si(L^2)Cl$	8.29(s, 1H)	-	7.31-6.65(m, 8H)	1.43(s, 3H)	_
6.	$Me_2Si(L^2)_2$	8.31(s, 1H)	_	7.32-6.60(m, 8H)	1.23(s, 3H)	_
7.	HL <sup>3</sup>	8.34(s, 1H)	4.31(s, 1H)	8.17-7.02(m, 8H)	_	2.47(s, 3H)
8.	Me <sub>2</sub> Si(L <sup>3</sup> )Cl	8.40(s, 1H)	_	8.21-7.12(m, 8H)	1.50(s, 3H)	2.35(s, 3H)
9.	$Me_2Si(L^3)_2$	8.41(s, 1H)	_	8.19–7.12(m, 8H)	1.20(s, 3H)	2.33(s, 3H)
10.	$HL^4$	8.25(s, 1H)	3.73(s, 1H)	8.11-6.56(m, 8H)	_	_
11.	$Me_2Si(L^4)Cl$	8.30(s, 1H)	-	8.30-7.20(m, 8H)	1.41(s, 3H)	_
12.	$Me_2Si(L^4)_2$	8.29(s, 1H)	_	8.21-7.32(m, 8H)	1.32(s, 3H)	_

TABLE 4 <sup>13</sup>C NMR spectral data ( $\delta$ , ppm) of the ligands [HL<sup>1</sup>, HL<sup>3</sup>] and their metal complexes

Sr. no.	Ligand/complex	Azomethine C-atom	Si-CH <sub>3</sub>	$C_1C_7$	$C_2C_8$	$C_3C_9$	$C_4C_{10}$	$C_5C_{11}$	$C_{6}C_{12}$
1.	$HL^1$	167.12	_	138.26	142.57	126.19	125.93	145.48	121.99
				146.92	136.33	130.62	125.80	126.19	121.89
2.	$HL^3$	168.33	_	138.70	12742	126.31	125.50	126.01	121.72
				146.36	135.85	130.95	125.07	126.57	121.63
3.	$Me_2Si(L^1)Cl$	160.57	19.50	138.13	143.12	126.31	125.79	145.31	121.19
				146.33	136.14	130.24	123.11	126.01	120.94
4.	$Me_2Si(L^1)_2$	160.63	19.78	138.11	143.76	126.15	125.83	145.21	121.15
				146.18	136.01	130.42	123.18	126.00	121.36
5.	Me <sub>2</sub> Si(L <sup>3</sup> )Cl	153.44	20.52	136.19	140.95	126.43	125.80	125.89	121.21
				148.55	135.33	130.17	124.62	126.79	121.18
6.	$Me_2Si(L^3)_2$	153.11	20.62	134.28	141.06	126.11	125.91	125.92	121.33
	· /-			145.92	135.52	130.25	124.74	126.69	121.52



carbon atoms attached with the azomethine nitrogen in the ligands appeared at  $\delta$  167.12 ppm[HL<sup>1</sup>] and  $\delta$  168.33 ppm[HL<sup>3</sup>]. However in the spectra of the corresponding silicon complexes, these bands shifted to lower  $\delta$  values (Shielding) and appeared at  $\delta$  160.57, 160.63, 153.44, 153.11 ppm, respectively, in 1:1 and 1:2 metal complexes. The considerable shift in the carbon atom attached to azomethine nitrogen indicates the involvement of nitrogen atom in coordination with silicon atom. New signals in the spectra of metal complexes from  $\delta$  19.50 to 20.62 ppm are assigned to methyl group attached to silicon. The signals due to different aromatic carbon atoms appeared in the range  $\delta$ 146.92–121.18 ppm, given in Table 4.

# <sup>29</sup>Si NMR Spectra

The <sup>29</sup> Si NMR spectra reflect the coordination number of the nucleus in the corresponding metal complexes.<sup>[21,25]</sup> In order to confirm the geometry of the complexes, <sup>29</sup> Si NMR spectra were recorded. The spectra show in each case only a sharp singlet indicating the formation of a single species. The <sup>29</sup> Si NMR spectra of Me<sub>2</sub>Si(L<sup>1</sup>)Cl and Me<sub>2</sub>Si(L<sup>2</sup>)Cl give sharp signals at  $\delta$  -96.24,  $\delta$  -97.38 ppm and the spectra of Me<sub>2</sub>Si(L<sup>1</sup>)<sub>2</sub> and Me<sub>2</sub>Si(L<sup>2</sup>)<sub>2</sub> give sharp signals at  $\delta$  -106.78,  $\delta$  -108.32 ppm, which indicates the penta and hexacoordinated environment, respectively, around the silicon atom.

Thus, on the basis of the above spectral features and molecular weight determination, it is suggested that the monomeric nature of the newly formed organosilicon complexes and trigonal bipyramidal and octahedral geometries have been proposed for 1:1 and 1:2 complexes, respectively, as shown in Figure 2.

# **BIOLOGICAL ASSAY**

The antimicrobial activity of newly synthesized ligands viz.  $(HL^1)$ ,  $(HL^4)$  and their complexes with Me<sub>2</sub>SiCl<sub>2</sub> have been screened against bacterial and yeast strains.

#### **TEST MICROORGANISMS**

Bacillus subtilis, Bacillus megaterium, Staphylococcus aureus, Pseudomonas aeruginosa, Escherichia coli, Saccharomyces cerevisiae, and Candida albicans.



FIG 2. Structure of silicon complexes, where HL<sup>1</sup>: 2-Chloro-5-nitrobenzylidene-o-aminothiophenol; HL<sup>2</sup>: 3-Bromobenzylidene-o-aminothiophenol; HL<sup>3</sup>: 4-Methylbenzylidene-o-aminothiophenol; and HL<sup>4</sup>: 2-Chlorobenzylidene-o-aminothiophenol.

Compounds	B. subtilis	B. megaterim	Staphylococcus aureus	Pseudomonas aerginosa	Escherichia coli	Saccharomyces cerevisiae	Candida albicans
$HL^1$	10	10	_	_	_	_	_
$HL^4$	15	20	20	_	15	_	_
$Me_2Si(L^1)Cl$	10		15	_	_	_	_
$Me_2Si(L^1)_2$	12	10	_	_	12	12	_
$Me_2Si(L^4)Cl$	_	10	_	_	12	12	-
$Me_2Si(L^4)_2$	-	10	-	-	12	12	_

TABLE 5 In vitro antimicrobial activity of chemically synthesized compounds (Zone inhibition in mm)

(-) means no activity

# Primary Screening

The antimicrobial activities of newly synthesized compounds were evaluated by using agar-well diffusion method with microbial strains. 20 mL of agar media was poured into each Petri plates and plates were swabbed with broth culture of 100  $\mu$ L of the respective microorganisms. 8 mm diameter wells were bored in the seeded agar plates and a 100  $\mu$ L volume of 1.0 mg/mL of each compound reconstituted in the DMSO was added into the well. The all plates were incubated at 37°C for 24h. Antimicrobial activities were determined by measuring the inhibition zone diameter. All the tests were made in triplicate and mean of the diameter of inhibition zone was calculated. The antimicrobial activities of the compounds were compared with Ciprofloxacin as standard. Zone of inhibition were determined for the compounds, and results are shown in Table 5.

# **Determination of Minimum Inhibitory Concentration** (MIC) and Minimum Bactericidal Concentration (MBC)

A minimum inhibitory concentration (MIC) is the lowest concentration of an antimicrobial compound that will inhibit the visible growth of a microorganism after overnight incubation. Minimum inhibitory concentrations are important in diagnostic laboratories to confirm resistance of microorganisms to an antimicrobial agent and also to monitor the activity of new antimicrobial agents. The minimum inhibitory concentration (MICs) of the chemically synthesized compound, viz. HL<sup>4</sup> (Table 6), was tested against bacterial and yeast strains through a macro dilution tube assay.<sup>[26]</sup> In this method, the test concentrations of chemically synthesized compound HL<sup>4</sup> were made from 128 to 0.25  $\mu$ g/mL in the sterile tubes no.1–11. Mueller Hinton Broth (MHB) medium was prepared. 100  $\mu$ L sterile MHB

were poured in each sterile tube and then 200  $\mu$ L compound was added in tube 1. Two fold serial dilutions were carried out from tube 1 to tube 11 and excess broth (100  $\mu$ L) was discarded from last tube no 11. 0.5 McFarland standard was made that contained 10<sup>8</sup> cells/mL. To each tube, 100  $\mu$ L of standard inoculum was added. Positive control (Containing inoculum but no compound) and negative control (containing compound but no inoculum) were made for the comparison. Ciprofloxacin was used as a standard drug. All the tubes were incubated for 24h at 37°C. A minimum bactericidal concentration (MBCs) is the lowest concentration of antimicrobial agents that will prevent the growth of an organism after subculture on to antibiotic free media.<sup>[26]</sup> Minimum bactericidal concentrations (Table 7) were determined by spreading the 100  $\mu$ L compound from one below MIC and four above MIC tubes on the sterile antibiotic free MHA plate. All the tubes were incubated for 24h at 37°C. The growth was observed on each plate.

#### **Biological Results and Discussion**

The compound HL<sup>1</sup> showed antibacterial activity against Bacillus species (B. megaterium and B. subtilis) in the form of zone of growth inhibition (10 mm). There was no activity against other microorganisms. The compound HL<sup>4</sup> showed antibacterial activity against E. Coli (15 mm), B. megaterium (20 mm), B. subtilis (15 mm), Staphylococcus aureus (20 mm) and did not show any activity against Pseudomonas aeruginosa, Saccharomyces cerevisiae and Candida albicans. The compound  $Me_2Si(L^1)Cl$  exhibited antimicrobial activity only against B. subtilis (10 mm) and Staphylococcus aureus (15 mm). The compound MeSi(L<sup>1</sup>)<sub>2</sub> showed antimicrobial activity against Bacillus megaterium (10 mm), B. subtilis (12 mm), Escherichia coli (12 mm) and Saccharomyces cerevisiae (12 mm). The compounds

	Minimum inhibitory concentrations (MIC) of compound							
		Microbial	strains					
Compound	Bacillus subtilis	B. megaterium	Escherichia coli	Saccharomyces cerevisia				
HL <sup>4</sup> Ciprofloxacin	128 2	128 2	64 4	64 2				

TABLE 6

	Minimum ba	ctericidal concent	trations (MBC) of c	compound
		Microbia	l strains	
Compound	Bacilus subtilis	B. megaterium	Escherichia coli	Saccharomyces cerevisiae
HL <sup>4</sup>	>128	>128	128	128

 TABLE 7

 Minimum bactericidal concentrations (MBC) of compound

HL<sup>1</sup>: 2-Chloro-5-nitrobenzylidene-o-aminothiophenol

HL<sup>2</sup>: 3-Bromobenzylidene-o-aminothiophenol

HL<sup>3</sup>: 4-Methylbenzylidene-o-aminothiophenol

HL4: 2-Chlorobenzylidene-o-aminothiophenol

 $Me_2Si(L^4)Cl$  showed almost same antimicrobial activity against *Escherichia coli* (12 mm), *Bacillus megaterium* (10 mm), and *Saccharomyces cerevisiae* (12 mm). The results implicate that *Pseudomonas aeruginosa* and *Candida albicans* were resistant to all the compounds evaluated in this experiment.

The MIC against *B. subtilis* and *B. megaterium* was  $128\mu$ g/mL and 64  $\mu$ g/mL against *E. coli* and *S. cerevisiae*. The MBC was greater than 128  $\mu$ g/mL against *B. subtilis* and *B. megaterium* and 128  $\mu$ g/mL against *E. coli* and *S. cerevisiae*. Compound HL<sup>4</sup> showed maximum activity in terms of zones of growth inhibition as compared to other compounds screened in this test. Hence, after testing *in vivo* compound HL<sup>4</sup> may be used in food and pharmaceutical industries as it inhibits *Escherichia coli*, *Bacillus megaterium*, *B. subtilis*, and *Staphylococcus aureus*, which are food borne in nature and the organisms *Pseudomonas aeruginosa*, *Saccharomyces cerevisiae*, and *Candida albicans*, which are harmful human pathogens.

The antibacterial results evidently showed that the activities of the ligands became more pronounced and significant when coordinated to the metal ions. It has been suggested that coordination reduces the polarity of the metal ion mainly because of partial sharing of its positive charge with donor group within the whole chelate ring system. This process of chelation thus increases the lipophilic nature of the central metal atom, which in turn, favors its permeation through the lipoid layer of the membrane thus causing the metal complex to cross the bacterial membrane more effectively and increasing the activity of the complexes. Besides from this, many other factors, such as solubility and dipole moment, conductivity influenced by metal ion may be possible reasons for remarkable antibacterial activities of these complexes.<sup>[27]</sup> It also has been observed that some moieties, such as azomethine linkage or heteroaromatic nucleus, introduced into such compounds exhibit extensive biological activities that may be responsible for the increase in hydrophobic character and liposolubility of the molecules in crossing the cell membrane of the microorganism and enhance biological utilization ratio and activity of complexes.<sup>[28]</sup>

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