

# Synthesis and Characterization of Silatranes Derived From Diethoxydichlorosilane

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**ABSTRACT:** The synthesis of three new silatranes using Schiff bases and diethoxydichlorosilane as encapsulating agent is described. Structures of these silatranes have been established by elemental analyses, infrared spectroscopy as well as  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectroscopy.

**KEYWORDS:** Diethoxydichlorosilane, schiff base, silatranes, salicylaldehyde, glyoxal

## I. INTRODUCTION

Coordination chemistry of Schiff base ligands has been a fascinating area of current research interest [1-8]. Depending on the respective coordination number and electronic properties of the functional ligands, many of these complexes display novel structural features or reactivity blueprints which give rise to various discoveries during the past decades [9]. Hypervalent silicon complexes [10-13] with Schiff bases have been broadly studied due to their bacteriostatic [14], antimicrobial [15], fungicidal [16,17], biocidal [18], bactericidal [19,20] properties. Here, we report the synthesis and characterization of higher coordinated silicon complexes derived from diethoxydichlorosilane and Schiff bases.

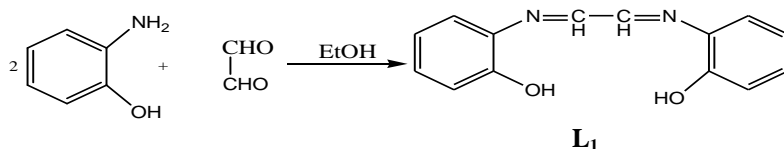
## II. MATERIAL AND METHODS

All reactions were carried out under nitrogen atmosphere using vacuum glassline. Solvent was freshly distilled under an inert atmosphere from phosphorus pentoxide (acetonitrile) before use. Triethylamine (Aldrich), Glyoxal (Qualigens), Salicylaldehyde (CDH), Hydrazine monohydrate (CDH), Benzene-1,2-diamine (CDH) and o-aminophenol (CDH) were used as supplied. Diethoxydichlorosilane was prepared using the standard method.

Infrared spectra were obtained as Nujol mulls and KBr pellet on a Perkin-Elmer RX-I FT IR spectrophotometer.  $^1\text{H}$  (300 MHz),  $^{13}\text{C}$  (75.45 MHz) NMR spectra were recorded on a Jeol and Bruker FT NMR (AL 300 MHz) spectrometer. Chemical shifts in ppm were determined relative to internal standard DMSO and  $\text{CDCl}_3$  and external standard tetramethylsilane (TMS). C, H and N analysis were obtained on a FLASH-2000 organic elemental analyzer.

### Reaction of Diethoxydichlorosilane with ligand ( $\text{L}_1$ )

Diethoxydichlorosilane (0.50mL) was added dropwise to a stirred solution of ( $\text{L}_1$ ) (0.63g, 2.64mmol) in acetonitrile (50 mL) and triethylamine. The reaction mixture was allowed to stir for 4h. The resulting orange coloured product (**1**) was filtered and dried under vacuum. M. pt: charring above  $240^\circ\text{C}$ ; Yield: 82%; Anal. Calc. for  $\text{C}_{18}\text{H}_{20}\text{N}_2\text{O}_4\text{Si}$  (Mol mass 356) : C:60.67; H: 5.61; N: 7.86; Si: 7.86; Found: C:61.09; H: 4.87; N: 8.09; Si:7.57; IR (Nujol, KBr,  $\text{cm}^{-1}$ ):  $\nu = 588$  ( $\text{N}\rightarrow\text{Si}$ ), 1070 ( $\text{Si-O(C)}$ ), 1270( $\text{C-O}$ ), 1594 ( $\text{C=N}$  azomethine);  $^1\text{H}$  NMR (DMSO):  $\delta = 1.21$  (6H, t,  $\text{CH}_3$ ); 3.66 (4H, q,  $\text{OCH}_2$ ); 6.66-7.15 (8H, m, aromatic); 7.49 (2H, s,  $\text{CH=N}$ );  $^{13}\text{C}$  NMR (DMSO):  $\delta = 18.36$  (2C,  $\text{CH}_3$ ); 40.73 (2C,  $\text{OCH}_2$ ); 116.53-121.68 (12C, aromatic); 95.99 (2C,  $\text{CH=N}$ ).



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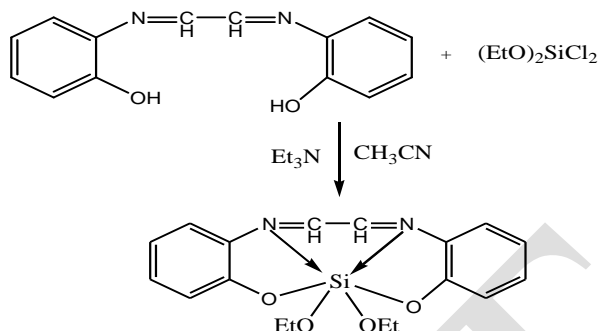


Figure 1 : Synthesis of Silatrane

### Reaction of Diethoxydichlorosilane with ligand (L<sub>2</sub>)

Diethoxydichlorosilane (0.50 mL) was added dropwise to a stirred solution of (L<sub>2</sub>) (0.63g, 2.64mmol) in acetonitrile (50 mL) and triethylamine. The reaction mixture was allowed to stir for 4h. The resulting orange coloured product (2) was filtered and dried under vacuum. M. pt: charring above 240°C; Yield: 82%; Anal. Calc. for C<sub>18</sub>H<sub>20</sub>N<sub>2</sub>O<sub>4</sub>Si (Mol mass 356) : C:60.67; H: 5.61; N: 7.86; Si: 7.86; Found: C:61.09; H: 4.87; N: 8.09; Si:7.57; IR (Nujol, KBr, cm<sup>-1</sup>):  $\nu$  = 586 (N→Si), 1074 (Si-O(C)), 1235(C-O), 1606 (C=N azomethine); <sup>1</sup>H NMR (DMSO):  $\delta$  = 1.30 (6H, t, CH<sub>3</sub>); 3.53 (4H, q, OCH<sub>2</sub>); 6.77-7.98 (8H, m, aromatic); 10.17 (2H, s, CH=N); <sup>13</sup>C NMR (DMSO):  $\delta$  = 8.10 (2C, CH<sub>3</sub>); 45.26 (2C, OCH<sub>2</sub>); 115.83-159.79 (12C, aromatic); 165.56 (2C, CH=N)

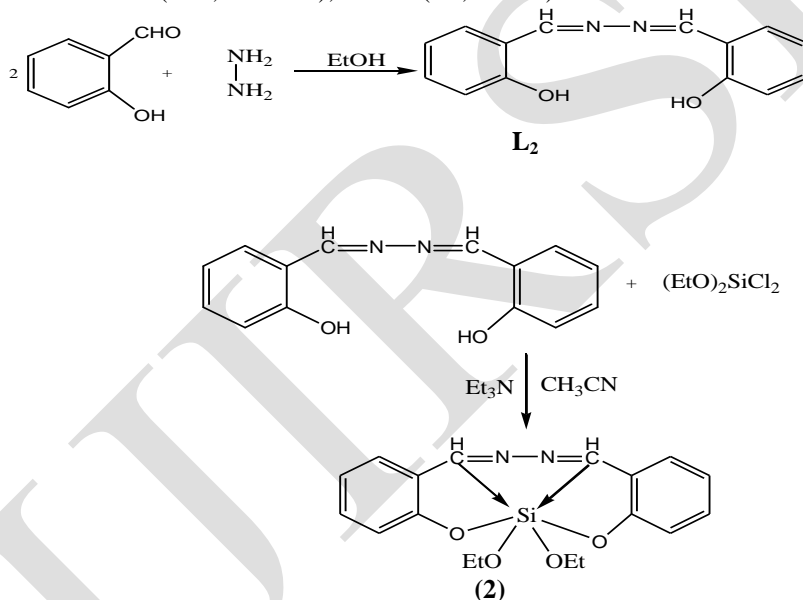


Figure 2 : Synthesis of Silatrane (2)

### Reaction of Diethoxydichlorosilane with ligand (L<sub>3</sub>)

Diethoxydichlorosilane (0.449g, 2.64mmol) was added dropwise to a stirred solution of (L<sub>3</sub>) (0.83g, 2.64mmol) in acetonitrile (50 mL) and triethylamine. The reaction mixture was allowed to stir for 4h. The resulting light yellow coloured product (3) was filtered and dried under vacuum. M. pt: charring above 240°C; Yield: 82%; Anal. Calc. for C<sub>24</sub>H<sub>24</sub>N<sub>2</sub>O<sub>4</sub>Si (Mol mass 432) : C:66.66; H: 5.55; N: 6.48; Si: 6.48; Found: C:66.34; H: 4.87; N:6.16; Si:6.78; IR (Nujol, KBr, cm<sup>-1</sup>):  $\nu$  = 573 (N→Si), 1026 (Si-O(C)), 1310(C-O), 1610 (C=N azomethine); <sup>1</sup>H NMR (DMSO):  $\delta$  = 1.28 (6H, t, CH<sub>3</sub>); 3.53 (4H, q, OCH<sub>2</sub>); 7.38-7.87 (12H, m, aromatic); 8.70 (2H, s, CH=N); <sup>13</sup>C NMR (DMSO):  $\delta$  = 17.98 (2C, CH<sub>3</sub>); 56.64 (2C, OCH<sub>2</sub>); 108.39-157.09 (18C, aromatic); 160.79 (2C, CH=N).

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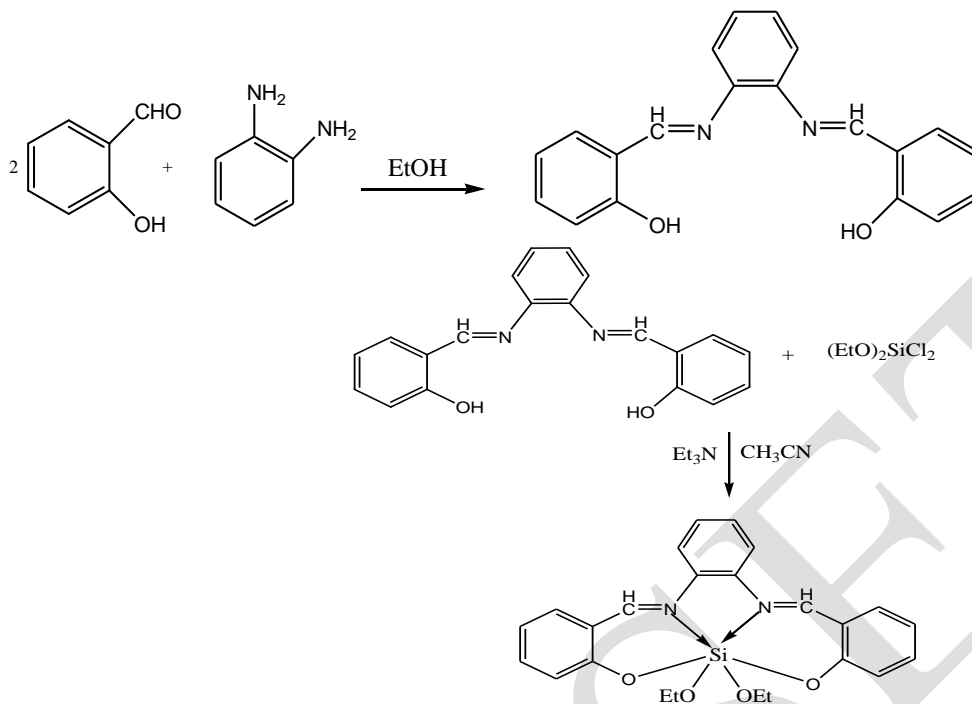


Figure 3 : Synthesis of Silatrane (3)

### III. RESULTS AND DISCUSSION

#### Syntheses

The new higher coordinated silicon complexes ( $C_{18}H_{20}N_2O_4Si$ ) (1), ( $C_{18}H_{20}N_2O_4Si$ ) (2) and ( $C_{24}H_{24}N_2O_4Si$ ) (3) were synthesized by the reaction of diethoxydichlorosilane with Schiff base ligands ( $L_1$ ), ( $L_2$ ) and ( $L_3$ ) as depicted in Scheme 1, Scheme 2 and Scheme 3 respectively. The composition of complexes was confirmed by their analytical data and the structures were suggested by spectroscopic investigations.

#### IR Spectra

The IR spectra of all the compounds were recorded in the range  $4000-400\text{ cm}^{-1}$ . The absorption frequencies were in accordance with the structure of the prepared compounds. The absorption bands of interest were those of  $N\rightarrow Si$ ,  $Si-O$ ,  $C-O$  and  $C=N$  bonds. IR spectrum in Nujol/KBr plates showed  $\nu N\rightarrow Si$ ,  $\nu Si-O$ ,  $\nu C-O$  and  $\nu C=N$  absorptions in the ranges  $573-588$ ,  $1026-1074$ ,  $1235-1310$  and  $1594-1610\text{ cm}^{-1}$  respectively for silatranes 1, 2 and 3.

#### NMR Spectra

Multinuclei ( $^1H$ ,  $^{13}C$ ) NMR spectra were consistent with the structure of the synthesized compounds. In  $^1H$  NMR spectra signals corresponding to  $-OCH_2$  and  $-CH_3$  of ethoxy groups appeared in the range  $3.53-3.66$  and  $1.21-1.30$  ppm respectively. The aromatic protons of the synthesized silatranes appeared in the region  $6.66-7.98$  ppm. The singlet of azomethine protons of complexes 1, 2 and 3 appeared at  $7.49$ ,  $10.17$  and  $8.70$  ppm respectively. The downfield shift of the azomethine proton in the silicon complexes indicated shielding which suggested that the most probable binding of the ligand with the silicon was realized through the nitrogen atom of the azomethine group.

In  $^{13}C$  NMR spectra signals corresponding to  $-OCH_2$  and  $-CH_3$  of ethoxy groups appeared in the range  $40.73-56.64$  and  $8.10-18.36$  ppm respectively. The aromatic region appeared at  $108.39-159.79$  ppm. The azomethine carbon of complex 1 appeared upfield ( $95.99$  ppm) as compared to complexes 2 and 3 ( $165.56$  and  $166.79$  ppm respectively). This may be because of direct linkage of the azomethine carbon to the aromatic rings in silatranes 2 and 3.

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## V. CONCLUSION

To examine the versatility of the formation of higher coordinate silicon compounds via silicon-nitrogen donor action, we carried out new synthesis employing the reaction of Schiff base ligands with diethoxydichlorosilane. The novel silatranes formed were successfully characterized.

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