SYNTHESIS AND CHARACTERIZATION OF ORGANOTIN (IV) COMPLEXES OF TRIDENTATE SCHIFF BASES CONTAINING -ONN/ONS DONOR ATOMS

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Synthesis and characterization of organotin(IV) complexes of tridentate Schiff bases containing –ONN/ONS donor atoms

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This project is submitted in partial fulfillment of the requirements for the Degree of Bachelor of Science with Honours (Resource Chemistry)

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DECLARATION

No portion of the work referred to in this report has been submitted in support of an application for another degree of qualification of this or any other university or institution of higher learning.

____________________________
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Synthesis and characterization of organotin(IV) complexes of tridentate Schiff bases containing –ONN/ONS donor atoms

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ABSTRACT

Four organotin(IV) complexes of methyl pyruvate thiosemicarbazone ligand (1) with \( R_nSnCl_{4-n} \) \((n = 1, 2)\) have been synthesized in the presence of base and refluxing methanol in 1:1:1 mole ratio (metal:base:ligand). Another four organotin(IV) complexes of salicylaldehyde 2-pyridine hydrazone ligand (2) in 1:1:1 mole ratio (metal:base:ligand) have also been synthesized with same method. All organotin(IV) complexes (3-10) have been characterized using CHN analyser, UV-Visible and IR spectral studies. All organotin(IV) complexes (3-10) are non electrolytic in nature. Spectral studies suggested that both the ligands (1-2) act as a mononegative tridentate nature in their organotin(IV) complexes. Therefore, a six coordinated molecular structure has been proposed for all the mentioned organotin(IV) complexes.

Key words: Organotin(IV) complexes; methyl pyruvate thiosemicarbazone; salicylaldehyde 2-pyridine hydrazone; spectral studies

ABSTRAK

Empat kompleks organotin(IV) dari ligan (1) metil piruvat tiosemikarbazon dengan \( R_nSnCl_{4-n} \) \((n = 1, 2)\) telah disintesis dengan kehadiran bes serta direfluk dalam methanol dengan nisbah molar 1:1:1 (logam:bes:ligan). Empat kompleks organotin(IV) dari ligan (2) salisilaldehid-2-piridin hidrazon dengan nisbah molar 1:1:1 (logam:bes:ligan) juga telah disintesis menggunakan kaedah yang sama. Kesemua kompleks organotin(IV) (3-10) telah dicirikan dengan menggunakan analisis CHN, UV-Visible dan IR kajian spektra. Kesemua kompleks organotin(IV) (3-10) bersifat bukan elektrolit secara semulajadi. Kajian spektra menunjukkan bahawa kedua-dua ligan (1-2) bertindak sebagai mononegatif tridentat secara semulajadi dalam kompleks organotin(IV). Lantaran itu, molekul struktur dengan enam koordinatan telah dicadangkan untuk kompleks organotin(IV) tersebut.

Kata kunci: Kompleks organotin(IV) metil piruvat tiosemikarbazon, salisilaldehid-2-piridin hidrazon, kajian spectra.
1.0 Introduction

1.1 Schiff base ligands and their organotin(IV) complexes

Schiff bases are an important class of ligands in metal coordination chemistry even after almost a century since their discovery (Pettinari et al., 2001).

Schiff base is a chemical compound containing a carbon-nitrogen double bond. The nitrogen atom is connected to an aryl or an alkyl group but not hydrogen. Schiff base can be synthesized by condensation reaction of primary amine and an aldehyde or ketone (scheme 1). The formation of a Schiff base is an important step in many biochemical reactions (Parker, 1993).

Reaction:

\[
\begin{align*}
R' - R' & \quad \text{H}_2\text{O} \\
\text{C} = \text{O} + R - \text{NH}_2 & \quad \text{R'} \quad \text{N} - \text{R} + \text{H}_2\text{O}
\end{align*}
\]

Scheme 1: Preparation of Schiff base

On the other hand, organotin(IV) complexes are compounds that have at least one tin-carbon bond exists which have the +4 oxidation state. Organotin(IV) compounds usually have the general formula of \(R_4\text{Sn}, R_3\text{SnX}, R_2\text{SnX}_2, \text{RSnX}_3\), where \(R\) is an alkyl or phenyl and \(X\) is an inorganic substituent, commonly chloride, fluoride, oxide, hydroxide, carboxylate or thiolate (Parker, 1993).

Organotin(IV) compounds have numerous applications (Pettinari et al., 2001). Among others, they are commercially used as agricultural biocides due to their antifungal properties. More recently, diorganotin(IV) compounds are being investigated for their antitumor activity.
Increasing attention has been devoted to Schiff base complexes of organotin(IV) moieties in view of their potential application in medicinal chemistry and biotechnology (Singh, H. L and Varshney, A.K., 2006; Pettinari et al., 2001; Singh., 1998; Belwal et al., 1997).

However, studies on the characteristics of Schiff base ligands containing -ONN/ONS donor atoms and their organotin(IV) complexes have not yet sufficient and deserves further, and deeper research. In review of the various interaction possibilities of the Schiff base ligands containing -ONN/ONS donor atoms with organotin(IV) chloride(s), the author has undertaken this project for the purpose of synthesizing and characterizing organotin(IV) complexes with the proposed Schiff base ligands containing -ONN/ONS donor atoms.

1.2 Objectives

The objectives of this research are:

(i) To synthesize tridentate Schiff base ligands containing -ONN/ONS donor atoms.
(ii) To synthesize organotin(IV) complexes with tridentate Schiff base ligands (ONN/ONS).
(iii) To characterize the Schiff base ligands (ONN/ONS) and their organotin(IV) complexes by CHN analyses, UV-Visible and FTIR spectra studies.
(iv) To determine the molar conductivity of the synthesized organotin(IV) complexes with Schiff base ligands (1-2).
2.0 Literature Review

2.1 Schiff base ligands containing -ONN/ONS donor atoms

A new tridentate ligand containing -ONN donor atom has been reported by Leovac (2004). Pyridoxal thiosemicarbazone has been synthesized from the condensation reaction of aqueous or alcoholic solutions of pyridoxal and the corresponding semicarbazide derivative. The structural formula of pyridoxal semicarbazone has shown as in Figure 1.

\[
\begin{align*}
\text{X=O} & \rightarrow \text{PLSC}, \text{X=S} & \rightarrow \text{PLTSC} \\
\end{align*}
\]

Figure 1: Structural formulas of pyridoxal semicarbazone

A tridentate ligand containing -ONS donor atom has been reported and shown as in Figure 2 (West et al., 1995).

Figure 2: Structure of 2-hydroxyacetophenone \(N(4)\)-substituted thiosemicarbazone
Yadav and his co-workers (2001) have synthesized the Schiff base ligand (Figure 3) by using 2:2 mole ratios of 2-hydroxyacetophenone, hydrazine hydrate and propylisothiocyanate.

![Figure 3: Structure of 2-hydroxyacetophenone N(3)-propylthiosemicarbazone](image)

Another type of Schiff base ligands with an -ONS donor atom has been synthesized and characterized (Ferrari et al., 2001). The general scheme of the reaction has shown as in Figure 4.

![Figure 4: The general scheme of the Schiff base ligands containing -ONS donor atoms.](image)
ohn and his co-workers (2005) have synthesized the Schiff base ligands (Figure 5) by using 1:1 mole ratio of 4-methyl \(N\) (4)-disubstituted thiosemicarbazone and 2-hydroxyacetophenone and respective amine.

![Diagram of ligand structures](image)

Figure 5: Structure of 2-hydroxyacetophenone \(N\) (4)-disubstituted thiosemicarbazone.
Another type of -ONS donor ligand has been reported by Pramanik (2004). A few metal complexes with potentially tridentate -ONS donor Schiff base of S-benzyl and S-methyl thiocarbazates with salicylaldehyde has been synthesized and characterized. The Figure six 6) below has shown the Schiff base ligand in thione and thiol form.

![Figure 6: Schiff base ligand containing -ONS donor atoms](image)

A Schiff base ligand containing -ONS donor atoms (Figure 7) has been synthesized by using 2:2 mole ratio of 2-hydroxyacetophenone and ethyl thiosemicarbazide in 10 mL methanol (Papathanasis et al., 2004).

![Figure 7: Structure of 2-hydroxyacetophenone ethyl thiosemicarbazone](image)
A new Schiff base ligand containing –ONN donor atoms has been reported (Affan et al., 2005) Figure 8).

![Structure of 2-acetylpyridinebenzhydrazone](image1)

Figure 8: Structure of 2-acetylpyridinebenzhydrazone

Josua and his co-workers (2001) have synthesized the Schiff base ligand containing –ONS donor atoms by using 1:1 mole ratio of -N (4)-phenylthiosemicarbazide and the 2-acetophenone in 15 mL ethanol.

![Structure of 2-hydroxyacetophenone-N (4)-phenyl thiosemicarbazone](image2)

Figure 9: Structure of 2-hydroxyacetophenone-N (4)-phenyl thiosemicarbazone
recently, Maurya and his co-workers (2006) have synthesized the Schiff base ligand containing –ONS donor atoms (Figure 10). The following structures are in thione and thiol form in solution.

![Thione form](image1)

![Thiol form](image2)

Figure 10: Structure of pyridoxal thiosemicarbazone
Some stable dimethyltin(IV) complexes of N-salicylidene derivatives of aroylhydrazine, S-methylhydrazinecarbodithionate and 4-substituted thiosemicarbazines have been prepared by the direct reaction of dimethyltin(IV) dichloride with sodium salts of the ligands in methanol. The formula structure of the complexes of dimethyltin(IV) dichloride with ligands, are shown in Figure 11 (Iskander et al., 1989).

![Figure 11: Formula structure of the dimethyltin(IV) complexes of N-salicylidene derivatives of aroylhydrazine, S-methylhydrazinecarbodithionate and 4-substituted thiosemicarbazines.](image-url)
A report on the synthesis and characterization of organotin(IV) complexes with potentially bidentate 2-acetylpyridinebenzhydrazone have been reported (Affan et al., 2005). Molecular structures of new organotin(IV) complexes have been determined by X-ray crystallography (Figure 12).

![Structure of mono methyldichloro[(2-acetylpyridinebenzhydrazone) ONN (-2)]tin(IV)](image)

Figure 12: Structure of mono methyldichloro[(2-acetylpyridinebenzhydrazone) ONN (-2)]tin(IV)

R = Me, Ph
Hunlin and his co-workers (2004) have been synthesized polymeric dibutyltin(IV) complexes with (2-pyrimidylthio) acetic acid, only the carboxylic acid group involve in the coordination to tin(IV) (Figure 13).

![Figure 13: Structure of polymeric di-n-butyltin(IV) complexes](image)

The synthesis and characterization of dimethyltin(IV) complexes with potentially tridentate 2-hydroxyacetophenone-\(N\) (4)-phenylthiosemicarbazone have been reported by Sousa and his co-workers (2001) (Figure 14).

![Figure 14: Structure of dimethyl [(2-hydroxyacetophenone-\(N\) (4)-phenylthiosemicarbazone) ONS (-2)] tin (IV)](image)
0 Materials and Methods

1 Experimental

All chemicals were purchased from Fluka, Aldrich and J.T. Baker. All solvents were dried and distilled by standard methods and stored in molecular sieves (4Å) (Amergo and Perrin, 1996). All the reactions were carried out under dry nitrogen atmosphere using Schlenk vacuum Line techniques.

2 Measurement

Schiff base ligands and their organotin(IV) complexes were characterized by elemental analyser namely CHN analyser, UV-Visible and FTIR spectral studies. Most of the organotin(IV) halide(s) and their organotin(IV)-Schiff base complexes were preserved in freezer for long-term storage and stability.

Infrared spectra were recorded as KBr disc using Perkin Elmer Spectrum GX Fourier-Transform Spectrometer (4000 – 400 cm⁻¹) at UNIMAS. Electronic absorption spectra were recorded with a suitable solvent on a Perkin Elmer Lambda 25 UV-Visible spectrometer at UNIMAS. Molar conductances were measured at room temperature using Hanna EC 215 conductivity meter at UNIMAS. Elemental analyses were recorded by using elemental analysers UNIMAS (Flashea 1112 series) and Microanalytical Lab of University of Dhaka Carlo Erba model 1108), Bangladesh.
3 Synthesis of Schiff base ligand (1)

3.1 Synthesis of methyl pyruvate thiosemicarbazone ligand \([\mathrm{C}_5\mathrm{H}_9\mathrm{N}_3\mathrm{O}_2\mathrm{S}]\) (1) containing ONS donor atoms

A mixture of methyl pyruvate (1.02g, 0.01 mole) and thiosemicarbazone (0.91g, 0.01 mole) in absolute ethanol (25 mL) were stirred and heated under refluxed for 3 hours. The reaction mixture was allowed to cool to room temperature for 30 minutes. The white precipitate formed was filtered off, and washed several times using absolute ethanol. The crystalline white solid obtained was purified by recrystallization from hot ethanol and dried in vacuo over silica gel. Yield = 1.87g, 85%, m.p 137-139 °C.

![Reaction Scheme](image)

Figure 15: Structure of methyl pyruvate thiosemicarbazone ligand (1)
3.2 Synthesis of $[\text{Me}_2\text{SnCl(C}_3\text{H}_8\text{N}_3\text{O}_2\text{S})]$ (3)

An anhydrous methanolic solution of ligand (1) (0.35g, 0.002 mole) was added by stirring to a potassium hydroxide solution (0.11g, 0.002 mole) under nitrogen atmosphere. The colour of the solution changed from off-white to yellow. The resulting mixture was stirred with constant stirring and refluxed for one hour and a solution of $\text{Me}_2\text{SnCl}_2$ (0.44g, 0.002 mole) in dry ethanol (10 mL) was added dropwise. The resulting solution was refluxed for 4-5 hours and allowed it to cool to room temperature. The precipitated potassium chloride (KCl) was removed by filtration and the filtrate was evaporated to dryness. The light yellow powder washed with hexane and dried in vacuo over silica gel. The organotin(IV) complex (3) was obtained as light yellow micro-crystals after recrystallized with methanol. Yield = 0.28g, 1.2%, m.p. 185-187 °C.
3 Synthesis of $[\text{Bu}_2\text{SnCl}(\text{C}_3\text{H}_8\text{N}_2\text{O}_2\text{S})]$ (4)

The complex (4) was synthesized by a similar method to that given in complex (3), with butyltin(IV) dichloride (0.608 g, 0.002 mole) being used instead of dimethyltin(IV) chloride. The organotin(IV) complex (4) was obtained as light yellow micro-crystals after crystallized with methanol-hexane (1:1 ratio). Yield = 0.61 g, 63.5%, m.p. 145-148 °C.

Figure 17: Proposed structure of organotin(IV) complex (4)