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

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Synthesis and Characterization of Organotin(IV) Complexes of Tridentate Schiff Bases Containing -ONO-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 -ONO-donor Atoms

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ABSTRACT

Six organotin(IV) complexes of salicylaldehyde-carbohydrazone ligand (1) with \( R_n\text{SnCl}_{4-n} \) (\( n = 1, 2 \)) have been synthesized in the presence of base and refluxing methanol in 1:2:1 molar ratio (metal:base:ligand). Two diorganotin(IV) complexes of salicylaldehyde-L-valine ligand (2) in 1:2:1 molar ratio (metal:base:ligand) have also been synthesized with same method. All organotin(IV) complexes (3-10) have been characterized using CHN analysis, 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 dinegative tridentate nature in their organotin(IV) complexes. Therefore, a five coordinated molecular structure has been proposed for all the mentioned organotin(IV) complexes.

Key words: Organotin(IV) complexes, salicylaldehyde-carbohydrazone, salicylaldehyde-L-valine, spectral studies.

Enam kompleks organotin(IV) dari ligan (1) salisilaldehid-karbohidrazon dengan \( R_n\text{SnCl}_{4-n} \) (\( n = 1, 2 \)) telah disintesis dengan kehadiran bes serta direfluks dalam methanol dengan nisbah molar 1:2:1 (logam:bes:ligan). Dua kompleks diorganotin(IV) dari ligan (2) salisilaldehid-L-valin dengan nisbah molar 1:2:1 (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 dinegatif tridentat secara semulajadi dalam kompleks organotin(IV). Lantaran itu, molekul struktur dengan lima koordinaten telah dicadangkan untuk kompleks organotin(IV) tersebut.

Kata kunci: Kompleks organotin(IV), salisilaldehid-karbohidrazon, salisilaldehid-L-valin, 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).

Any of the class of derivatives of aldehydes or ketones with primary amines will give stable and well-characterized imines through the condensation reaction. When one or both of the reactants is aromatic, the imine is known as Schiff base (Parker, 1993).

Schiff bases may coordinate with the central metal ion or atom either in keto or anionic (enol) forms via the loss of ‘amide’ hydrogen (Affan et al., 2003). Organotin(IV) compounds are formed when the compounds that have at least one tin-carbon bond exists which have the +4 oxidation state. Organotin(IV) compounds usually have the general formula $R_4Sn$, $R_3SnX$, $R_2SnX_2$, and $RSnX_3$, 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 and antibacterial properties (Ghosh et al., 2005). More recently diorganotin(IV) compounds are being investigated for their antitumor activity (Affan et al., 2003). Increasing attention has been devoted to Schiff base complexes of organotin(IV) moieties in view of their potential applications in medicinal chemistry and biotechnology (Pettinari et al., 2001; Singh, 1998; Belwal et al., 1997).
However, studies on the characteristic of Schiff base ligands containing ONO-donor atoms and their organotin(IV) complexes have not yet sufficient and deserves further, and deeper research. In view of the various interaction possibilities of the Schiff base ligands containing ONO-donor atoms with organotin(IV) chloride(s), efforts have been undertaken for the purpose of synthesizing and characterizing of the organotin(IV) complexes with Schiff base ligands containing ONO-donor atoms.

1.2 Objectives

The purposes of this research are:

1.2.1 To synthesize tridentate Schiff base ligands containing ONO-donor atoms.

1.2.2 To synthesize organotin(IV) complexes with tridentate Schiff base (ONO) ligands.

1.2.3 To characterize the Schiff base (ONO) ligands and their organotin(IV) complexes by CHN analysis, UV-Visible and FTIR spectral analyses and also determine molar conductance values of organotin(IV) complexes.
2.0 Literature review

2.1 Schiff base ligands containing ONO donor atoms

A tridentate dibasic Schiff base ligand with an ONO donor set reacts with \((\text{n-C}_4\text{H}_9)_4\text{NVO}_3\) in the presence of a small amount of acetic acid to form a monomeric and five-coordinated oxovanadium(V) complexes of the type \([\text{VO(L)}(\text{OCH}_2\text{CH}_3)]\) \((H_2L = \text{N-benzoylacetonem-bromobenzoylhydrazone})\) (Wang et al., 1996). A biological effect of the ligand and its complex has been studied. The complex shows more inhibition than the ligand.

Another type of -ONO- tridentate ligand has been reported by Hassaan (1997). A few metal complexes of isatin-isonicotinoylhydrazone has been synthesized and characterized. The isatin-isonicotinoylhydrazone is derived from isatin and isonicotinic acid hydrazide, as in Figure 1 below.

![Fig. 1: Isatin-isonicotinoylhydrazone.](image)

Two isomerisc ONO Schiff bases donor ligands (I and II) derived from salicylaldehyde, 2-hydroxy-1-naphthaldehyde and o-hydroxybenzylamine or o-aminobenzylalcohol have been prepared and characterized (Maurya et al., 1997) (Figure 2).
Dey and his co-workers (1999) have synthesized the Schiff base (Figure 3) by using 1:1 molar ratio of anthranilic acid and salicylaldehyde in methanol.

![Figure 3: N-(2-carboxyphenyl)salicylideneimine.](image)

Fig. 3: N-(2-carboxyphenyl)salicylideneimine.
A tridentate ligand, 2-(o-hydroxyphenyliminomethyl)pyridine N-oxide, has been synthesized by the condensation reaction of 2-pyridinecarboxaldehyde N-oxide with 2-aminophenol as shown in Figure 4 (Baran et al., 2002).

![Figure 4](image_url)

Fig. 4: 2-(o-hydroxyphenyliminomethyl)pyridine N-oxide.

Another type of novel ONO-donor Schiff bases N-salicylidene-\(n\)-alkanoylhydrazines derived from \(n\)-alkanoylhydrazines and salicylaldehyde in methanol by Iskander and his co-workers (2003) (Figure 5).

![Figure 5](image_url)

\[
\text{Fig. 5: } N\text{-salicylidene-}n\text{-alkanoylhydrazines.}
\]

\[n = 0, 4, 5, 6.\]
Recently, a novel tridentates Schiff base \([\text{ONO}]\) donor ligand has been synthesized by Chen and his co-workers (2005) (Figure 6).

![ONO-Schiff base ligand](image)

Fig. 6: ONO-Schiff base ligand.

A new tridentate ONO-ligand derived from salicylaldehyde and 2-aminophenol containing the ONO donor set of atoms (Barba et al., 2005), which is shown in Figure 7.

![Tridentate ONO-Schiff base ligand](image)

Fig. 7: Tridentate ONO-Schiff base ligand.

2.2 Schiff base ligands containing ONO donor atoms and their organotin(IV) complexes

Some stable dimethyltin(IV) complexes of N-salicylidene derivatives of aroylhydrazine, S-methylhydrazinecarbodithionate and 4-substituted thiosemicarbazines have been prepared by the 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, which are formed by condensation reactions (Iskander et al., 1989), are shown in Figure 8.
Fig. 8: Formula structure of the dimethyltin(IV) complexes of N-salicylidene derivatives of aroylhydrazine, S-methylhydrazinecarbodithionate and 4-substituted thiosemicarbazines.

A report on the synthesis and characterization of some mono- and dimethyltin(IV) complexes with potentially tridentate N-salicylidene- and N-pyridoxylidene acylhydrazines have been prepared by Labib and his co-workers (1996). The reported structure for the organotin(IV) complexes are monochloromonomethyl[(N-salicylidene acylhydrazinato)ONO(-2)]tin(IV), dimethyl[(N-salicylidene acylhydrazine)ONO(-2)]tin(IV), monomethylmonochloro[(N-pyridoxylidene acylhydrazine)ONO(-2)]tin(IV), and dimethyl[(N-pyridoxylidene acylhydrazine)ONO(-2)]tin(IV) which are shown in Figure 9 to 12.

Fig. 9: Monochloromonomethyl[(N-salicylidene acylhydrazinato)ONO(-2)]tin(IV).
Fig. 10: Dimethyl[(N-salicylidene acylhydrazine)ONO(-2)]tin(IV).

Fig. 11: Monomethylmonochloro[(N-pyridoxyliene acylhydrazine)ONO(-2)]tin(IV).

Fig. 12: Dimethyl[(N-pyridoxylidene acylhydrazine)ONO(-2)]tin(IV).

From the interaction of 2-{{(2-hydroxyphenyl)imino[2-methyl]phenol(saloph2)} with tin(IV) and organotin(IV) acceptors, the derivatives [SnR3(saloph)] (R = Me or Bu), [SnR2(saloph)] (R = Me, Bu, Bu′, Vi or Ph), [SnRX(saloph)(solvent)] (R = Me, Bu, Ph or X; X = Cl, Br or I; solvent = CH₃OH or H₂O), [Sn(saloph)₂], [R₂SnCl₂(saloph₂)] (R = Me or Bu) have been obtained and characterized, as shown in Figure 13 (Pettinari et al., 2001).
Another research reported that two diorganotin(IV) complexes of the general formula $R_2Sn[Ph(O)C=CH-C(Me)=N-N=C(O)Ph]$ ($R=Ph,$ 1; $R=Me,$ 2), is shown in Figure 14. Both complexes have been synthesized from the corresponding diorganotin(IV) dichloride and the ligand [4-phenyl-2,4-butanedionebenzoylhydrazone(2-)], derived from benzoyl acetone and benzoyl hydrazide in methanol at room temperature in presence of triethylamine (Dey et al., 2003).

Fig. 14: Proposed structure of compounds (1) and (2).
3.0 Materials and Methods

3.1 Experimental

All chemicals were purchased from Fluka, Aldrich and J.T. Baker. All solvents were dried and distilled before used as follows: ethanol and methanol form magnesium turnings and iodine, then stored in molecular sieves (Armarego and Perrin, 1996). All the reactions were carried out under dry nitrogen atmosphere using Schlenk Vacuum line techniques.

3.2 Measurement

Schiff base ligands and their organotin(IV) complexes were characterized by elemental analysis namely CHN analysis, UV-Visible and FTIR spectra 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 Elemer Lambda 25 UV-Visible spectrometer at UNIMAS. Molar conductances were measured at room temperature using Hanna EC 215 conductivity meter at UNIMAS.
3.3 Synthesis of Schiff base ligand (1) and its organotin(IV) complexes (3-8)

3.3.1 Synthesis of salicylaldehyde-carbohydrazone ligand \([\text{C}_6\text{H}_5\text{N}_4\text{O}_2]\) (1) containing ONO donor atoms

A mixture of carbohydrazide (0.450 g, 0.005 mole) and salicylaldehyde (0.610 g, 0.005 mole) in absolute ethanol (25 mL) were heated under reflux for 3 hours. The reaction mixture was allowed to cool down 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 vacuum dessicator. Yield: 0.67 g, 62%, m.p. 196-198 °C.

\[
\text{H}_2\text{N}^+\text{N}^-\text{CO}^-\text{NH}_2 + \text{OHC}^-\text{Ph} \xrightarrow{\text{Ethanol} \quad \text{Stirred, refluxed} \quad 3 \text{ hours}} \text{H}_2\text{N}^+\text{N}^-\text{CO}^-\text{N}^-\text{C}^-\text{Ph} \quad \text{Ligand (1)}
\]

Fig. 15: Proposed structure of salicylaldehyde-carbohydrazone ligand (1).
3.3.2 Synthesis of [Me₂Sn(C₅H₈N₂O₂)]·H₂O (3)

Ligand (1) (0.388 g, 0.002 mole) in dry methanol (10 mL) was added by stirring to a potassium hydroxide solution (0.224 g, 0.004 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 dimethyltin(IV) dichloride (0.440 g, 0.002 mole) in dry methanol (10 mL) was added dropwise. The resulting solution was refluxed for 3 hours and allowed to cool down to room temperature. The precipitated potassium chloride was removed by filtration and the filtrate was evaporated to dryness, washed by hexane and dried in vacuum dessicator. The organotin(IV) complex (3) was obtained as light yellow micro-crystals by recrystallization from methanol-hexane (1:1 ratio). Yield: 0.43 g, 52%, m.p. 293-295 °C.

![Chemical structure](image)

**Fig. 16: Proposed structure of organotin(IV) complexes (3-5).**
3.3.3 Synthesis of [Bu$_2$Sn(C$_9$H$_8$N$_2$O$_2$)] (4)

The procedure described above for (3) was followed for the preparation of (4), with dibutyltin(IV) dichloride (0.608 g, 0.002 mole) was being used instead of dimethyltin(IV) dichloride. The organotin(IV) complex (4) was obtained as light yellow micro-crystals by recrystallization from methanol-hexane (1:1 ratio). Yield: 0.55 g, 55%, m.p. 212-214 °C.

3.3.4 Synthesis of [Ph$_2$Sn(C$_9$H$_8$N$_2$O$_2$)] (5)

Complex (5) was prepared similarly to complex (3), diphenyltin(IV) dichloride (0.688 g, 0.002 mole) was being used instead of dimethyltin(IV) dichloride. The organotin(IV) complex (5) was obtained as light yellow micro-crystals by recrystallization from chloroform-hexane (1:1 ratio). Yield: 0.61 g, 57%, m.p. 206-208 °C.
3.3.5 Synthesis of \([\text{MeSnCl}(C_8H_8N_4O_2)]\) (6)

The procedure described above for (3) was followed for the preparation of (6), with methyltin(IV) trichloride (0.481 g, 0.002 mole) being used instead of dimethyltin(IV) dichloride. The organotin(IV) complex (6) was obtained as light brown micro-crystals by recrystallization from methanol-hexane (1:1 ratio). Yield: 0.67 g, 77%, m.p. 216-218 °C.

\[
\text{H}_2\text{N} - \text{N} \equiv \text{C} - \text{H} + 2\text{KOH} \xrightarrow{\text{MeOH}, \text{N}_2} \text{Resulting ligand solution} + \text{RSnCl}_3
\]

Salicylaldehyde-carbohydrazone

\[
\text{R} = \text{Me} (6), \text{Bu} (7), \text{Ph} (8).
\]

Fig. 17: Proposed structure of organotin(IV) complexes (6-8).

3.3.6 Synthesis of \([\text{BuSnCl}(C_8H_8N_4O_2)]\) (7)

Complex (7) was synthesized in a similar way as in complex (3), using butyltin(IV) trichloride (0.565 g, 0.002 mole), instead of dimethyltin(IV) dichloride. The organotin(IV) complex (7) was obtained as light brown micro-crystals by recrystallization from methanol-hexane (1:1 ratio). Yield: 0.79 g, 83%, m.p. 148-150 °C.
3.3.7 Synthesis of [PhSnCl(C₆H₅N₂O₃)] (8)

The procedure described above for (3) was followed for the preparation of (8), with phenyltin(IV) trichloride (0.605 g, 0.002 mole) was being used instead of dimethyltin(IV) dichloride. The organotin(IV) complex (8) was obtained as light brown micro-crystals by recrystallization from methanol-hexane (1:1 ratio). Yield: 0.54 g, 54%, m.p. 298-300 °C.

3.4 Synthesis of Schiff base ligand (2) and its diorganotin(IV) complexes (9-10)

3.4.1 Synthesis of salicylaldehyde-L-valine ligand \([\text{C}_{12}\text{H}_{15}\text{NO}_3]\) (2) containing ONO donor atoms

A mixture of L-valine (0.585 g, 0.005 mole) and salicylaldehyde (0.610 g, 0.005 mole) in absolute ethanol (25 mL) were heated under reflux for 5 hours. The reaction mixture was allowed to cool down to room temperature for 30 minutes. The crystalline white solid was obtained after slowly evaporation of solvent, washed with ethanol and was finally purified by recrystallization from hot ethanol, and dried in vacuum dessicator. Yield: 0.75 g, 63%, m.p. 177-179 °C.

\[ \begin{align*}
\text{Salicylaldehyde} & \quad + \quad \text{L-valine} \\
\text{Stirred, refluxed 5 hours} & \quad \rightarrow \\
\text{Ethanol} & \quad \text{Ligand (2)}
\end{align*} \]

Fig. 18: Proposed structure of salicylaldehyde-L-valine ligand (2).