

Synthesis and characterization of organotin(IV) complexes with hydrazone/carbohydrazone
ligand

MARGARET ANAK PANGGAI

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

Faculty of Resource Science and Technology
UNIVERSITI MALAYSIA SARAWAK
2008

Acknowledgement

First and for most, I would like to praise the Lord because of His love and blessing I manage to finish my final year project. Then my many hearty thanks go out to Dr. Md. Abu Affan for being an all-around good supervisor, for the use of his time and his ideas from the first until the end of my project, and the Department of Chemistry for the support provided and knowledge. Also I would like to convey my heartiest appreciation to the Master students especially to Mr. Foo Siong Wan for his gracious encouragement and help with his expertise in FTIR and UV-Visible. Besides that, I am thankful to Ms. Irene Foo Ping Ping and my friends in the laboratory for being there to help out and making lab life fun. I would like to take this opportunity to express my deepest gratitude to my beloved mother Mdm. Christina Merai Anyeh and family Guan, Francis, and Lauriebeeld Igoh for all of their unconditional loves and supports. Finally, I would like to thank to Universiti Malaysia Sarawak, especially Faculty of Resource Science and Technology for providing the facilities.

TABLE OF CONTENT

ACKNOWLEDGEMENT	i
ABSTRACT	iv
LIST OF TABLES	v
LIST OF FIGURE	vi
CHAPTER 1 Introduction	
1.1 Hydrazone/carbohydrazone ligands and their organotin(IV) complexes	1
CHAPTER 2 Literature Review	
2.1 Hydrazone/carbohydrazone ligands	3
2.2 Organotin(IV) complexes with hydrazone/carbohydrazone ligands	9
CHAPTER 3 Objectives	12
CHAPTER 4 Materials and Methods	13
4.1 Experimental	13
4.2 Measurement	13
4.2.1 Solvent distillation	13
4.3 Synthesis of hydrazone/carbohydrazone ligands	
4.3.1 Synthesis of carbohydrazone- <i>bis</i> (benzaldehyde) [C ₁₅ H ₁₄ N ₄ O] (1)	14
4.3.2 Synthesis of benzophenone-salicylhydrazone [C ₂₀ H ₁₆ N ₂ O ₂] (2)	15
4.4 Synthesis of organotin(IV) complexes (3-10)	
4.4.1 Synthesis of [Bu ₂ SnCl(C ₁₅ H ₁₄ N ₄ O)] (3)	16
4.4.2 Synthesis of [Ph ₂ SnCl(C ₁₅ H ₁₄ N ₄ O)] (4)	17
4.4.3 Synthesis of [Me ₂ SnCl(C ₁₅ H ₁₄ N ₄ O)] (5)	17
4.4.4 Synthesis of [MeSnCl ₃ (C ₁₅ H ₁₄ N ₄ O)] (6)	17
4.4.5 Synthesis of [PhSnCl ₃ (C ₁₅ H ₁₄ N ₄ O)] (7)	18
4.4.6 Synthesis of [BuSnCl ₃ (C ₁₅ H ₁₄ N ₄ O)] (8)	19

4.4.7 Synthesis of [Ph ₂ SnCl(C ₂₀ H ₁₆ N ₂ O ₂)] (9)	19
4.4.8 Synthesis of [Me ₂ SnCl(C ₁₅ H ₁₄ N ₄ O)] (10)	20
CHAPTER 5 Results and Discussions	
5.1 General	21
5.2 Physical data	22
5.3 Spectroscopic studies of carbohydrazone ligand (1) and its organotin(IV) complexes (3-8)	
5.3.1 UV-Visible analyses of ligand (1) and its organotin(IV) complexes	24
5.3.2 IR spectra analyses of ligand (1) and its organotin(IV) complexes	26
5.4 Spectroscopic studies of hydrazone ligand (2) and its organotin(IV) complexes (9-10)	
5.4.1 UV-Visible analyses of ligand (2) and its organotin(IV) complexes	30
5.4.2 IR spectra analyses of ligand (2) and its organotin(IV) complexes	32
5.4.3 ¹ H NMR spectra of ligand (2) and its organotin(IV) complex (10)	36
CHAPTER 6 Conclusion	39
CHAPTER 7 Recommendation	40
CHAPTER 8 References	41
APPENDIX I	46
APPENDIX II	47
APPENDIX III	48
APPENDIX IV	49
APPENDIX V	50
APPENDIX VI	51

LIST OF TABLES

Table 1:	The physical and analytical data for ligands (1-2) and their organotin(IV) complexes (3-10)	22
Table 2:	Molar conductance value for organotin(IV) complexes (3-10) of ligands (1-2)	23
Table 3:	The λ_{max} (nm) peaks of ligand (1) and its organotin(IV) complexes (3-8)	24
Table 4:	Main IR data of carbohydrazone ligand (1) and its organotin(IV) complexes (3-8)	27
Table 5:	The λ_{max} (nm) peaks of ligand (2) and its organotin(IV) complexes (9-10)	30
Table 6:	Main IR data of hydrazone ligand (1) and its organotin(IV) complexes (9-10)	33

LIST OF FIGURES

Figure 1:	Formula structure of 2,6-diacetylpyridine <i>bis</i> (2-thenoylhydrazone) [X= S] and 2,6-diacetylpyridine <i>bis</i> (2-furanolhydrazone), [X=O]	3
Figure 2:	Formula structures of 2-acetylpyridine- <i>N</i> (4)-phenylthiocarbazono, HAP ₄ P, and 2-hydroxy-acetophenone- <i>N</i> (4)-phenylthiosemicarbazone, H ₂ DAP ₄ P	4
Scheme 1:	The keto and enol tautomers of pyridoxal thiosemicarbazone	4
Figure 3:	Structure of 2,9-diformylphenonethroline <i>bis</i> (benzoyl)hydrazone ligand	5
Figure 4:	Formula structure of LH ₃ (C ₁₆ H ₁₄ N ₂ O ₅)	5
Figure 5:	Formula structure of salicylidene hydrazone (SalBzGH)	6
Figure 6:	Structure for 2,6- <i>bis</i> (1-salicyloylhydrazonoethyl)pyridine [H ₄ daps]	7
Figure 7:	Pyruvic acid thiophene-2-carboxylic hydrazone	7
Figure 8:	Isomer of ketone-2,4-dinitrophenylhydrazones	8
Figure 9:	Proposed structures for sterol hydrazones Hpy1 and Hpy2	8
Figure 10:	Stannacyclohexyl and stannacycloheptyl derivatives of dipeptides (AA)	9
Scheme 2:	1,5- <i>bis</i> (isatin)thiocarbonohydrazone and of its <i>N</i> -alkyl derivatives	10
Figure 11:	<i>N</i> -alkylisatin thiosemicarbazones and its diorganotin(IV) complexes	10
Figure 12:	Structure of pyruvic acid 3-hydroxy-2-naphthoyl hydrazone	11
Figure 13:	Organotin(IV) complexes with ONO- donor ligand	11
Scheme 3:	Proposed structure of carbohydrazone- <i>bis</i> (benzaldehyde) ligand (1)	14
Scheme 4:	Proposed structure of Benzophenone-salicylhydrazone ligand (2)	15
Scheme 5:	Proposed structure of dimethyltin(IV) complex (3), dibutyltin(IV) complex (4) and diphenyltin(IV) complex (5) of ligand (1)	16
Scheme 6:	Proposed structure of methyltin(IV) complex (6), butyltin(IV) complex (7) and phenyltin(IV) complex (8) of ligand (1)	18
Scheme 7:	Proposed structure of dimethyltin(IV) complex (9) and diphenyltin(IV) complex (10) of ligand (2)	20
Figure 14:	UV spectra of carbohydrazone- <i>bis</i> (banzaldehyde) (1) and its organotin(IV)	

	complex (7) in DMF (1×10^{-4} M)	25
Figure 15:	IR spectrum of Carbohydrazone- <i>bis</i> (benzaldehyde) (1) (As KBr disc)	28
Figure 16:	IR spectrum of dimethyltin(IV) complex (5) of ligand (1) (As KBr disc)	29
Figure 17:	UV spectra of benzophenone-salicylhydrazone ligand (2) and its dimethyltin(IV) complex (10) in DMF (1×10^{-4} M)	31
Figure 18:	The IR spectrum for benzophenone-salicylhydrazone ligand (2) (As KBr disc)	34
Figure 19:	The IR spectrum of diphenyltin(IV) complex of ligand (2) (As KBr disc)	35
Figure 20:	^1H NMR spectra for benzophenone-salicylhydrazone (2) (CDCl_3)	37
Figure 21:	^1H NMR spectra for diphenyltin(IV) complex (10) (CDCl_3)	38

Synthesis and characterization of organotin(IV) complexes with hydrazone/carbohydrazone ligand

Margaret Anak Panggai

Department of Chemistry
Faculty of Resource Science and Technology
Universiti Malaysia Sarawak

ABSTRACT

Six organotin(IV) complexes of carbohydrazone-*bis*(benzaldehyde) ligand (**1**) with $R_n\text{SnCl}_{4-n}$ ($R = \text{Me, Bu and Ph, } n = 1, 2$) have been synthesized in refluxing methanol under N_2 atmosphere. Another, two organotin(IV) complexes of benzophenone-salicylhydrazone ligand (**2**) have also been synthesized with the same method but in the presence of base in refluxing methanol in 1:1 mole ratio. All organotin(IV) complexes (**3-10**) have been characterized by different physicochemical techniques like CHN analyses, UV-visible and IR spectral studies. Among them, ligand (**2**) and its diphenyltin(IV) complex (**9**) have also been characterized by ^1H NMR spectral analyses. All organotin(IV) complexes are non electrolytic in nature. Spectral studies suggest that ligand (**1**) act as bidentate nature while the ligand (**2**) act as a mononegative bidentate nature in their organotin(IV) complexes. Five- and six- coordinated molecular structures have been proposed for all the organotin(IV) complexes.

Key words: Organotin(IV) complexes; benzophenone-salicylhydrazone; carbohydrazone-*bis*(benzaldehyde); spectral studies.

ABSTRAK

Enam kompleks organotin(IV) dari ligan (**1**) carbohydrazone-*bis*(benzaldehyde) dengan $R_n\text{SnCl}_4$ ($R = \text{Me, Bu and Ph; } n = 1, 2$) telah disintesis dengan kehadiran bes dan telah direfluks di dalam metanol dengan nisbah 1:1 mol. Dua kompleks organotin(IV) dari ligan (**2**) benzophenone-salicylhydrazone) juga telah disintesis melalui kaedah yang sama. Semua kompleks organotin(IV) (**3-10**) telah dicirikan dengan menggunakan analisis CHN, UV-visible, dan juga kajian spektra IR. Semua kompleks organotin(IV) (**3-10**) bersifat bukan elektrolitik secara semulajadi. Salah satu kompleks iaitu diphenyltin(IV) kompleks (**9**) dan ligan (**2**) telah dicirikan menggunakan ^1H NMR. Melalui kajian spektra didapati bahawa ligan (**1**) bertindak sebagai bidentate secara semulajadi, manakala ligan (**2**) bertindak sebagai mononegatif bidentate dalam kompleks organotin(IV), dan oleh sebab itu struktur molekul yang mempunyai lima dan enam koordinatan telah dicadangkan untuk kompleks organotin(IV) ini.

Kata kunci: Kompleks organotin(IV); benzophenone-salicylhydrazone; carbohydrazone-*bis*(benzaldehyde); kajian spektra.

Chapter 1

Introduction

1.1 Hydrazone/Carbohydrazone ligands and their organotin(IV) complexes

The product of condensation reaction of hydrazine with an aldehyde or ketone is called hydrazone (Affan *et al.*, 2003). Hydrazone ligands have high capability to react as chelating agent to metal atoms or metal ions especially transition metals. The functional group of hydrazone $\text{H} \searrow \text{N} - \text{N} = \text{C} \swarrow$ belongs to a large class of azomethines which are distinguished from other members of this class for example imines, oximes by the presence of two adjacent N atoms. Hydrazone ligands that coordinate to metal ion via N, O or S atoms are called as ON- and NS- donor ligands. Hydrazones are known to have variety of applications such as in pharmaceutical industry as drugs for treatment of cancer, schizophrenia and leprosy (Mishra *et al.*, 2007).

The coordination chemistry of hydrazones is an extensive area of study, numerous transition metal complexes of these ligands have been investigated (Mehrota, 1998). In recent years, the hydrazones have been drawn much attention from coordination chemists, due to its strong tendency of aroyl hydrazones to chelate transition metals and main group metals (Mishra *et al.*, 2007).

Due to various type of coordinating capabilities and chemical applications, a number of new hydrazone ligands have been synthesized. Organotin(IV) complexes have been studied for their biomedical and commercial applications. There are more and more report on synthesis, anti-tumor activities and structural elucidation of various di-organotin derivatives (Yin and Chen, 2006).

Recently, the coordination chemistry of carbohydrazone ligand has been studied with organotin(IV) ions (Affan *et al.*, 2007). Hydrazone and their metal complexes with transition metals have provoked wide interest for their apparent biological and pharmaceutical activities. Some carbohydrazone ligands behaved as NN chelating agents in the neutral form and ONNO/NNO chelating agents in deprotonated form. Complexes of carbohydrazone with non-transition metal ions such as organotin(IV) have not received much attention (Affan *et al.*, 2007).

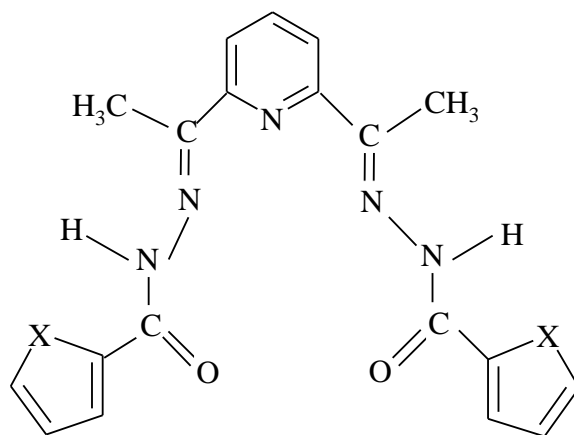
In spite of this, the focus of my research is on the synthesis and characterization of organotin(IV) complexes with hydrazone/carbohydrazone ligands.

Chapter 2

Literature Review

2.1 Hydrazone/Carbohydrazone ligands

De Sousa and his co-workers (1999) have reported the synthesis of some stable ligand, 2,6-diacetylpyridine *bis*(2-furanolhydrazone) and 2,6-diacetylpyridine *bis*(2-thenoylhydrazone) (Figure 1).



X = O, S

Figure 1: Formula structure of 2,6-diacetylpyridine *bis*(2-thenoylhydrazone) [X= S] and 2,6-diacetylpyridine *bis*(2-furanolhydrazone), [X=O]

De Sousa *et al.* (2001) have synthesized 2-acetylpyridine-*N*(4)-phenylthiocarbazonate, HAP₄P and 2-hydroxy-acetophenone-*N*(4)-phenylthiosemicarbazone, H₂DAP₄P, as well as their metal complexes with organotin(IV). These structures of the ligands are shown in Figure 2.

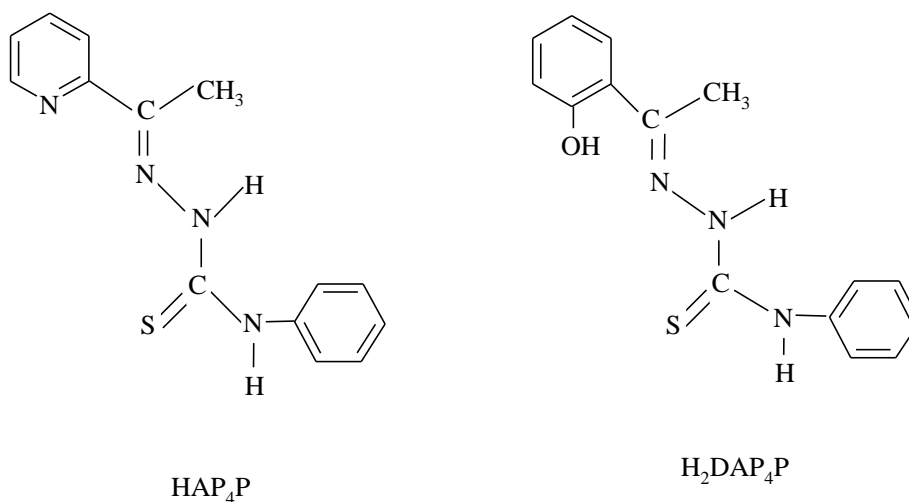
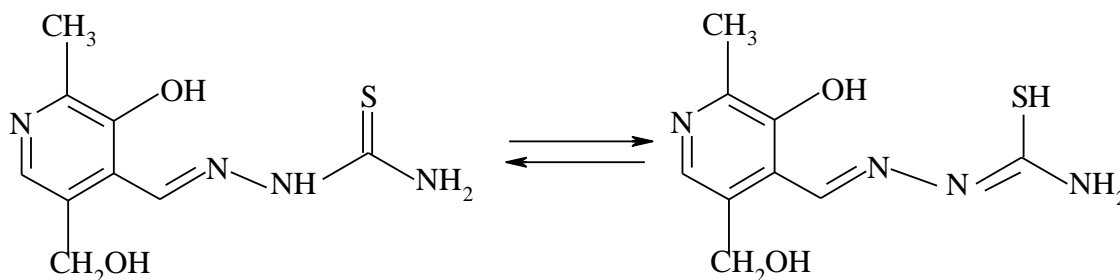


Figure 2: Formula structures of 2-acetylpyridine-*N*(4)-phenylthiocarbazonate, HAP₄P, and 2-hydroxy-acetophenone-*N*(4)-phenylthiosemicarbazone, H₂DAP₄P

In 2006, Maurya and his co-workers have synthesized the N, O-donor ligands derived from pyridoxal and thiosemicarbazide. The pyridoxal and thiosemicarbazide are widely used in the biological activity. Scheme 1 shows the thio and thiol tautomers of pyridoxal thiosemicarbazone.



Scheme 1: The thio and thiol tautomers of pyridoxal thiosemicarbazone

Carcelli *et al.* (2003) have reported the 2,9-diformylphenanthroline *bis*(benzoyl)hydrazone, with aim to obtain a potentially hexadentate ligand (Figure 3).

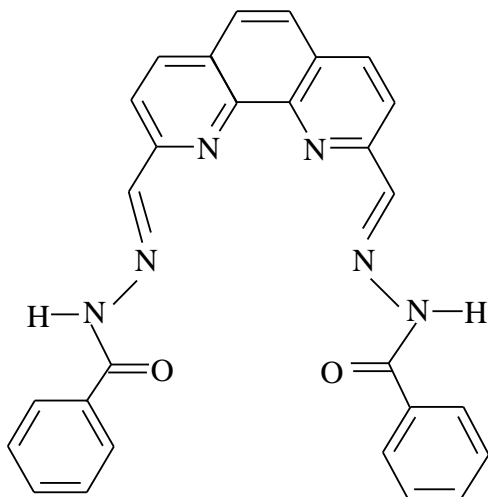


Figure 3: Structure of 2,9-diformylphenanthroline *bis*(benzoyl)hydrazone ligand

Baligar and Revankar (2006) have reported the synthesis of mononucleating hydrazone ligand [LH₃]. This ligand was synthesized by condensation reaction of salicyloylhydrazine and 2-formylphenoxy. Figure 4 shows the formula structure of mononucleating hydrazone ligand, LH₃.

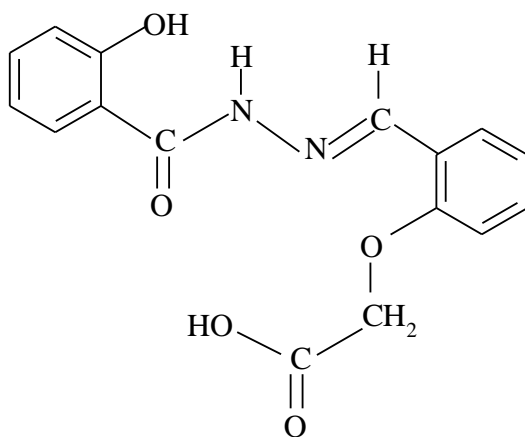


Figure 4: Formula structure of LH₃ (C₁₆H₁₄N₂O₅)

Lonibala *et al.* (2006) have reported the polidentate ligand known as salicylidene hydrazone (SalBzGH, Figure 5). It has two isomeric forms in the ambient temperature. However, it assumes only one configuration when coordinated to the lanthanide(III) ion, and prefers to form complexes only in the ionic form. Besides that, Lonibala *et al.* (2006) have also synthesized complexes of SalBzGH with transition metal ions.

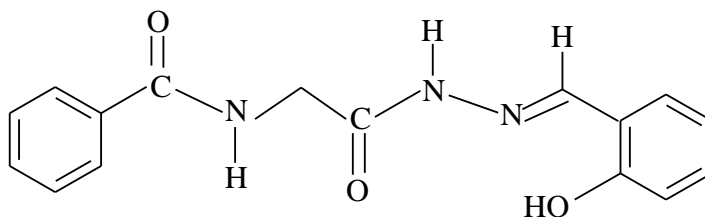


Figure 5: Formula structure of salicylidene hydrazone (SalBzGH)

According to Pedrido *et al.* (2006) the chelating properties of pentadentate hydrazone derivated ligands have been intensively studied during the last two decades. The variety of structures observed for this type of metal complex, their pharmacological activity and their magnetic properties make these compounds became a special interest.

Mono and polynuclear complexes have been obtained by changing the metal, the experimental conditions and the geometry of hydrazone ligands in order to increase their properties and coordinative ability. It is a very convenient route for the preparation of metallic complexes derived from the ligand 2,6-bis(1-salicyloylhydrazoneethyl)pyridine [H₄daps] by employing the electrochemical synthesis. Figure 6 shows the structure of 2,6-bis(1-salicyloylhydrazoneethyl)pyridine.

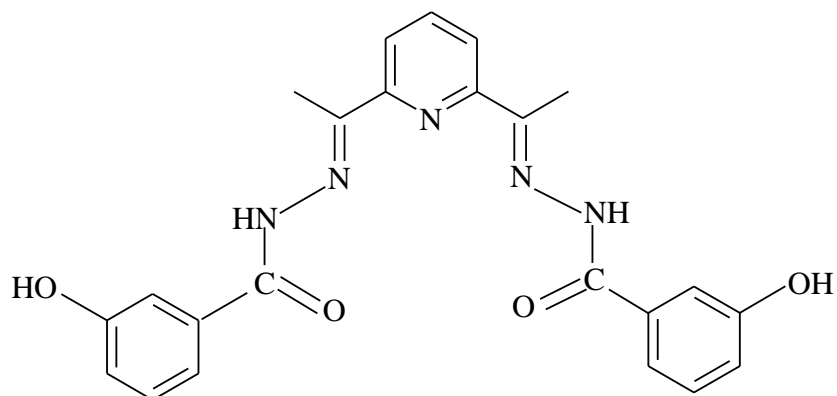


Figure 6: Structure for 2,6-bis(1-salicyloylhydrazoneethyl)pyridine [H₄daps]

Yin *et al.* (2007) have reported the synthesis and characterization of organotin(IV) compounds with pyruvic acid thiophene-2-carboxylic hydrazone. Figure 7 shows the structure of pyruvic acid thiophene-2-carboxylic hydrazone.

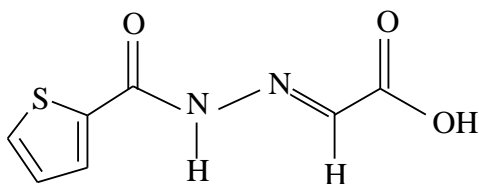


Figure 7: Pyruvic acid thiophene-2-carboxylic hydrazone

In 2007, Uchiyama *et al.* have described the isomerization mechanism of ketone-2,4-dinitrophenylhydrazones. According to their research, ketone-2,4-dinitrophenylhydrazones have two isomers which are *E*-isomer and *Z*-isomer. However purified carbonyl-2,4-dinitrophenylhydrazone demonstrated only the *E*-isomer. Figure 8 shows the isomer of ketone-2,4-dinitrophenylhydrazones.

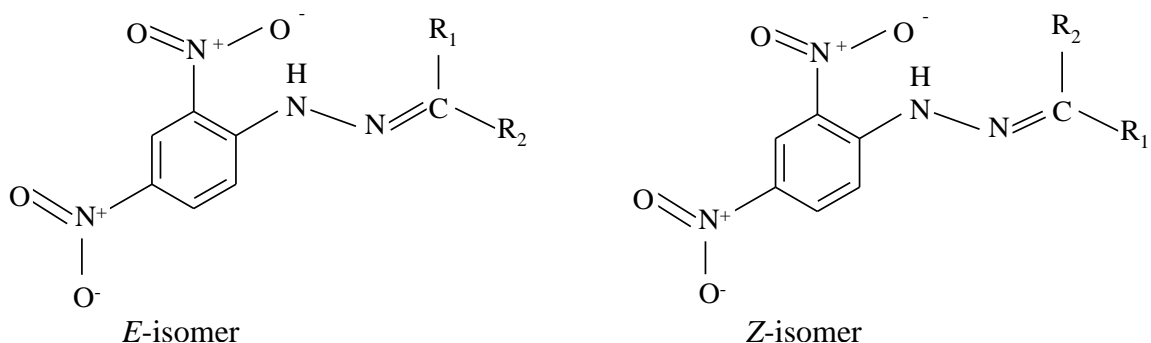


Figure 8: Isomer of ketone-2,4-dinitrophenylhydrazones

Recently, Visbal *et al.* (2008) have reported the synthesis and characterization of platinum-sterol hydrazone complexes with biological activity against *Leishmania (L.) mexicana*. Searching for a new drugs that combine high parasitocidal and low toxicity, Visbal and his co-workers, coordinated a sterol ligands to platinum(II). Figure 9 showed the proposed structure for the sterol hydrazones.

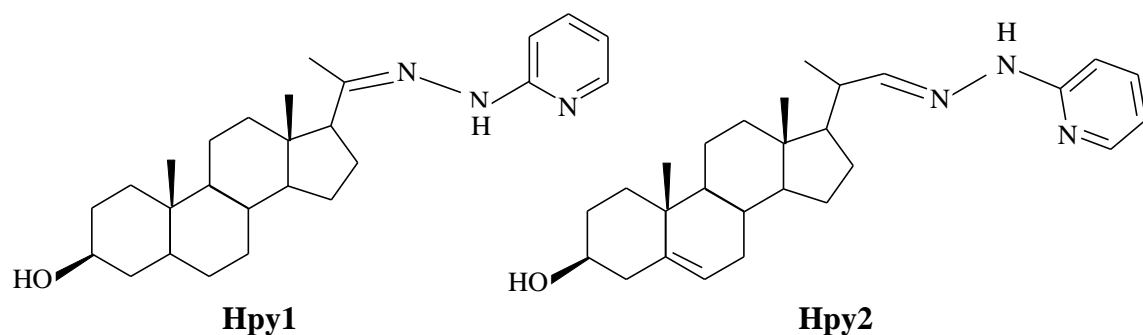


Figure 9: Proposed structures for sterol hydrazones Hpy1 and Hpy2

2.2 Organotin(IV) complexes with hydrazone/carbohydrazone ligands

It is well known that organotin(IV) compounds displayed strong biological activity. Most of the organotin(IV) compounds are generally very toxic, even though at low concentration. The biological activity is essentially determined by the number and nature of the organic groups bound to the central Sn(IV) atom. It seems that the nature of the anionic group is of only secondary importance (Pallerito and Nagy, 2002).

Pallerito and Nagy (2002) have prepared and investigated the stannacyclohexyl and stannacycloheptyl derivatives of dipeptides (AA). In these compounds, Sn is a member of a ring system (Figure 10). These solid compounds indicated that the AA acted as a tridentate {O, N, N} ligand and the Sn has a trigonal-bypiramidal environment. An analogous structure has been found for the undissociated molecules in methanol solutions. This work clearly demonstrated that there are no basic restrictions for Sn(IV) to extend its coordination sphere (at least to attain pentacoordination) when it is an integral part of a cycloalkyl system.

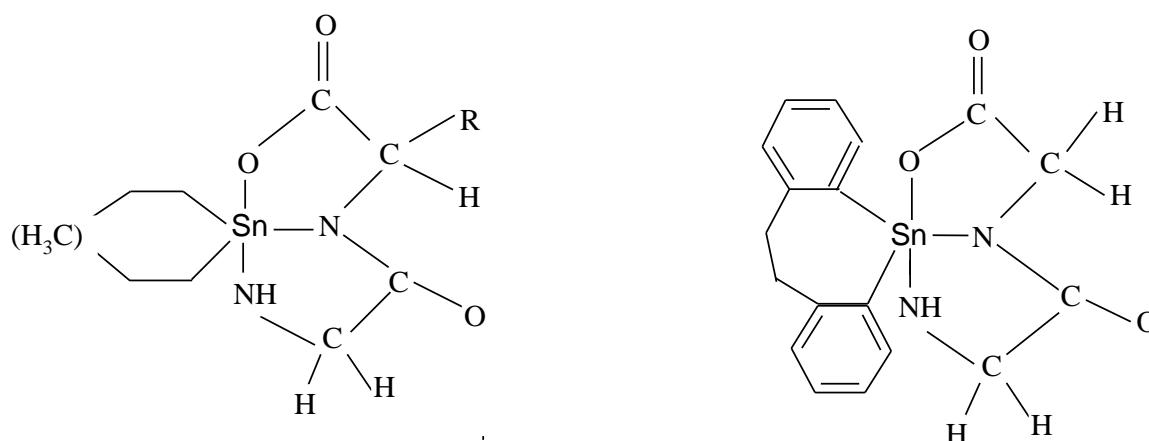
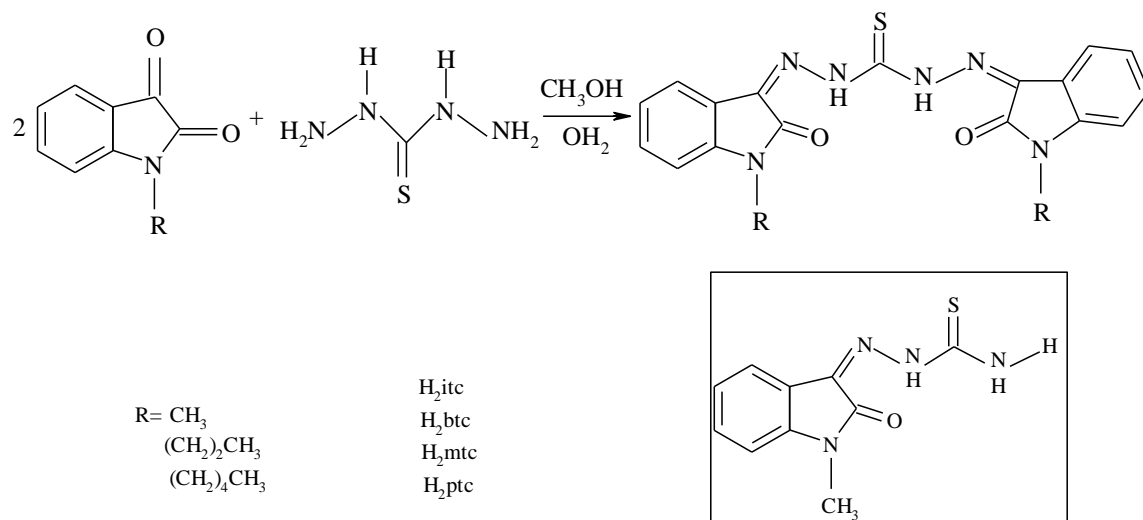


Figure 10: Stannacyclohexyl and stannacycloheptyl derivatives of dipeptides (AA)

Bacchi *et al.* (2005) have reported the synthesis as shown in scheme 2 and characterized of diorganotin(IV) complexes with 1,5-*bis*(isatin)thiocarbonohydrazone and its *N*-alkyl derivatives as shown in figure 11.



Scheme 2: 1,5-*bis*(isatin)thiocarbonohydrazone and of its *N*-alkyl derivatives

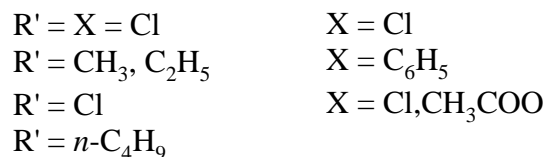
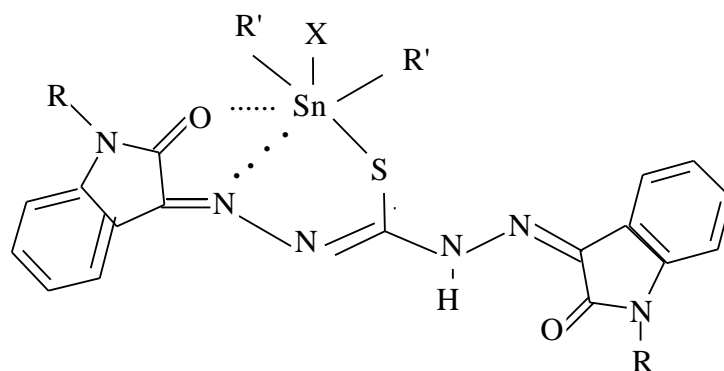


Figure 11: *N*-alkylisatin thiosemicarbazones and its diorganotin(IV) complexes

Yin and Chen (2006) have reported the synthesis of di- and tri-organotin(IV) complexes with Schiff base ligand pyruvic acid 3-hydroxy-2-naphthoyl hydrazone. Figure 12 shows the structure of the organotin(IV) complexes with hydrazone ligand.

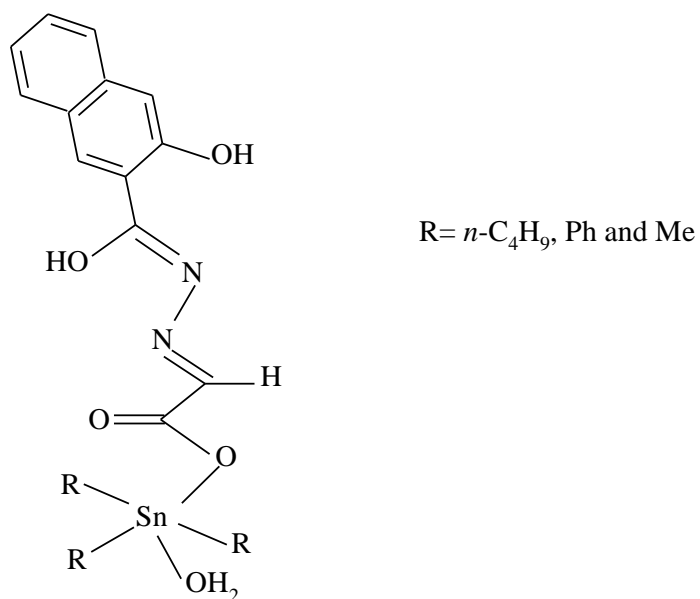


Figure 12: Structure of organotin(IV) complex with pyruvic acid 3-hydroxy-2-naphthoyl hydrazone

Affan *et al.* (2007) have reported the characterization of novel organotin(IV) complexes with carbohydrazone ligand containing ONO- donor ligand. Figure 13 showed the structure of the organotin(IV) complexes.

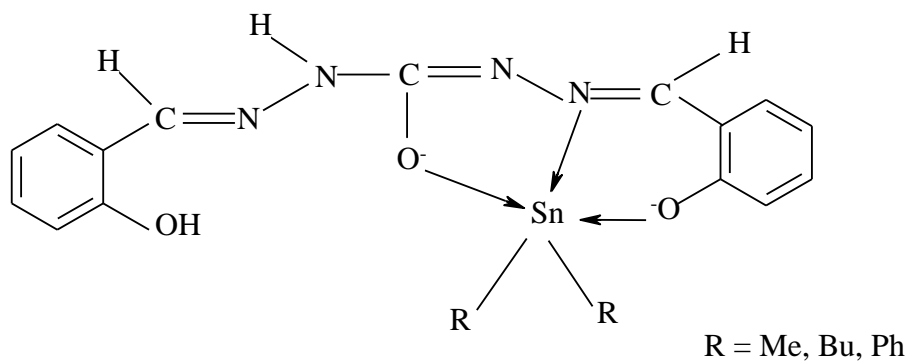


Figure 13: Organotin(IV) complexes with ONO- donor ligand

Chapter 3

Objectives

The main objectives of this project are:

- 1) to synthesize two hydrazone/carbohydrazone ligands.
- 2) to synthesize organotin(IV) complexes with hydrazone/carbohydrazone ligands.
- 3) to characterize organotin(IV)-hydrazone/carbohydrazone complexes, by elemental analyses, UV-visible, FTIR and ^1H NMR spectra analyses.
- 4) to determine the molar conductance values of the synthesized organotin(IV) complexes.

Chapter 4

Materials and Methods

4.1 Experimental

Hydrazone/carbohydrazone ligands namely carbohydrazone-*bis*(benzaldehyde) and benzophenone-salicylhydrazone have been synthesized by condensation reaction. The first ligand was prepared by the condensation reaction of carbohydrazide and benzaldehyde in 1:2 mole ratio. The second ligand was also has synthesized by condensation reaction of salicylhydrazide and benzophenone in 1:1 mole ratio in absolute ethanol. The organotin(IV) hydrazone/carbohydrazone complexes were synthesized by direct reaction of hydrazone/carbohydrazone ligand with the respective organotin(IV) halide(s) in the presence of base (Affan *et al.*, 2007).

4.2 Measurement

The research was conducted in Inorganic Research Laboratory at UNIMAS. All chemicals were purchased from Fluka, Aldrich or J.T. Baker. All the reactions were carried out under nitrogen atmosphere by using Schlenk Vacuum line techniques. Hydrazone/carbohydrazone ligand and their organotin(IV) complexes were characterized with Flash EA 1112 Series CHN elemental analyzer at UNIMAS. Infrared spectra were recorded as KBr disc using Perkin Elmer Spectrum GX Fourier-Transform Spectrometer ($4000 - 400 \text{ cm}^{-1}$). ^1H NMR spectra were recorded with CDCl_3 on Jeol 500 FT-NMR spectrophotometer. Electronic spectra will be recorded with DMF on Perkin Elmer Lambda 25 UV-Visible Spectrometer at UNIMAS. Molar conductances were measured at room temperature using Jenway 4510 conductivity meter at UNIMAS.

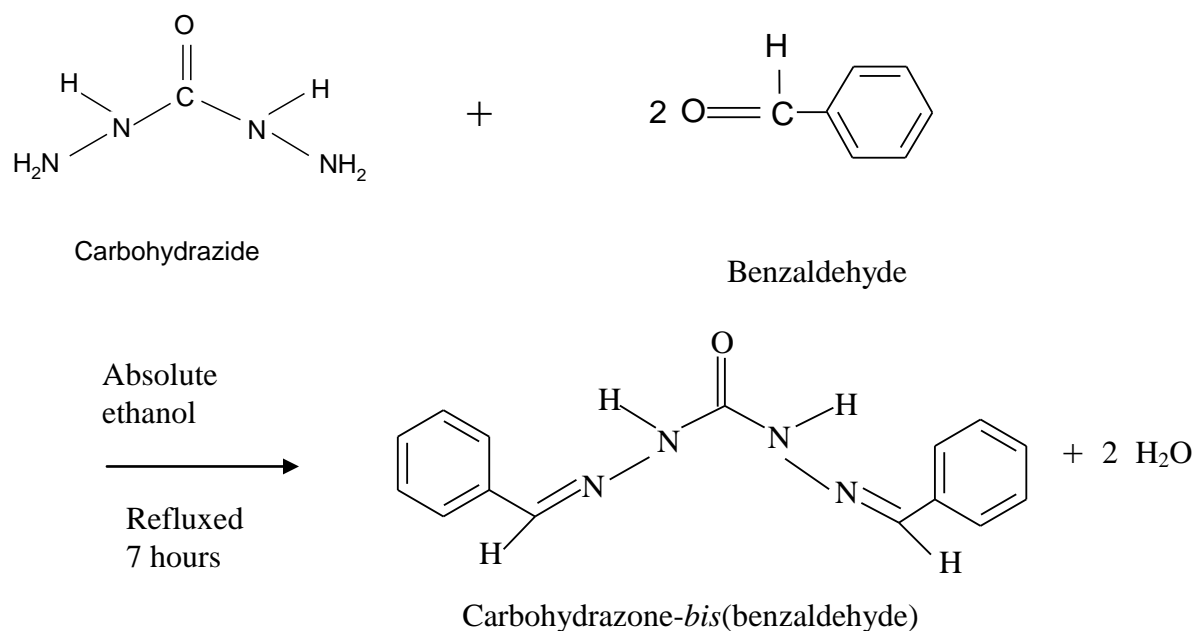
4.2.1 Solvent Distillation

All the solvents were distilled according to published methods by Armarego before used (Armarego *et al.*, 1996).

4.3 Synthesis of hydrazone/carbohydrazone ligands

4.3.1 Synthesis of carbohydrazone-*bis*(benzaldehyde) [C₁₅H₁₄N₄O] (1)

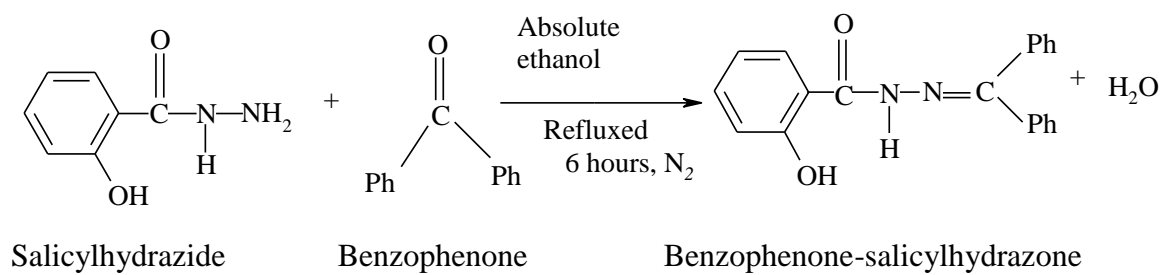
Carbohydrazide, (0.900g, 0.010 mole) and benzaldehyde (2.122 g, 0.020 mole) were dissolved in absolute ethanol (20 mL) with constant stirring and heated under reflux for 7 hours. This reaction mixture was allowed to cool to room temperature for 30 minutes. The white precipitate formed, filtered off and washed several times with absolute ethanol. The white crystals were collected and dried in *vacuo* over silica gel. Yield 0.921g, 64% m.p. 196-198 °C.



Scheme 3: Proposed structure of carbohydrazone-*bis*(benzaldehyde) ligand (1)

4.3.2 Synthesis of benzophenone-salicylhydrazone ligand [C₂₀H₁₆N₂O₂] (2)

A mixture of salicylhydrazide (0.456 g, 0.003 mole) and benzophenone (0.547 g, 0.003 mole) were dissolved in absolute ethanol (20 mL) with constant stirring and heated under reflux for 6 hours. This reaction mixture was allowed to cool to room temperature for 30 minutes. The white precipitate formed and filtered off, and washed several times by using absolute ethanol. The white crystalline solids were collected and dried in *vacuo* over silica gel. Yield 0.396g, 58%, m.p. 147-149 °C.

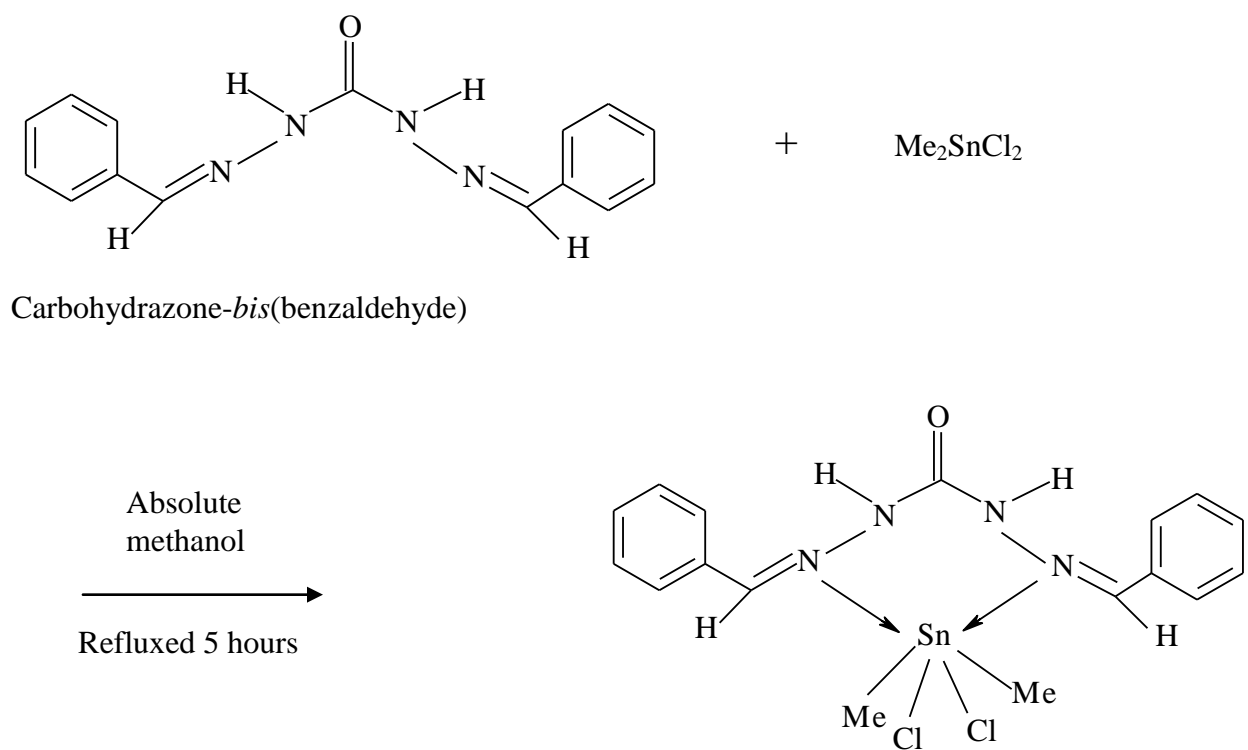


Scheme 4: Proposed structure of Benzophenone-salicylhydrazone ligand (2)

4.4 Synthesis of organotin(IV) complexes (3-10)

4.4.1 Synthesis of $[\text{Me}_2\text{SnCl}_2(\text{C}_{15}\text{H}_{14}\text{N}_4\text{O})]$ (3)

Complex (3) was synthesized using dimethyltin(IV) dichloride (0.219 g, 0.001 mole) . The dimethyltin(IV) dichloride dissolved in methanol (10 mL) was slowly added into ligand solution. After heated under reflux for 5 hours, the solution was evaporated to leave the solid precipitate. Crystalline solids were collected through filtration, washed it several times with absolute methanol and dried in silica gel. Yield 0.29g, 54%, m.p: 215-217 °C.



Scheme 5: Proposed structure of dimethyltin(IV) complex (3) with ligand (1)