

Ain shams University
University College for women
(Art, Science and Education)
Cairo, Egypt.

### Synthesis and Characterization of Some New Schiff Base Complexes Using 1,6 hexanediamine

Supervised by:

**APPROVED** 

Prof. Dr. Marguerite A. wassef

Prof. of Inorganic Chemistry

Prof. Dr. Ali Mostafa A. Hassan

Prof. of Inorganic Chemistry

Ass. Prof. Hoda Abd-El hakam bayoumi

Ass. Prof. of Inorganic chemistry

**APPROVED** 

Head of Chemistry Department Prof. Dr.



لسلاداب والعلوم والتربيسة قســـم الكيميـاء

(رسالة ماجستير)

: منارة أحمد محمد أبوب اسم الطالبة

عنوان الرسالة : تطيق و توسيف بعض المتراكبات البديدة لقاعدة شبغت وأستخدام 6,1 ثنائي الامين مكسان

: ماجستير في العلوم (كيمياء غير العضوية و التحليلية) اسم الدرجة

#### لجنة الاشراف

1- ا.د /مرجریت أنیس وادف

أستاذ الكيمياء نيرالعضوية — كلية البنائد – جامعة عين همس

2- ا د/ على مصطفى ملذ العرب

أستاذ الكيمياء غير العضوية – كلية العلوو– جامعة الازمر

3 – د/مدی نمید الحکم بیومی

أستاذ مساغد الكيمياء غير العضوية - كلية البنائد - جامعة غين شمس

رئيس قسم الكيمياء



جامعة عين شمس كلي التهادات التهادات والعلوم والتربية قسم الكيمياء

اسم الطالبــة: منارة أحمد محمد أيوب

الدرجة العلمية: بكالوريوس علوم

القسم التابع له: الكيمياء

اسم الكليه: البنات للاداب والعلوم والتربية

الجامع ـــة: عين شمس

سنة التخرج: مايو 2003

سنة المنح: 2008

# **QUALIFICATION**

**Student Name**: Manara Ahmed Mohammed Ayoub

**Scientific Degree:** B.Sc. (Chemistry)

**Department**: Chemistry

Name of Collage: University College for Women

(Arts, Science and Education)

**University**: Ain Shams

**B.Sc. Graduation Date**: May 2003

#### **NOTE**

# The candidate has attended courses for one year covering the following topics:

*	Advanced Chemical Reactions
*	Instrumental Analysis
*	Kinetics and Catalysis
*	Thermodynamics
*	Photo Chemistry
*	Quantum Chemistry
*	Advanced Course of Reaction Mechanism
*	Advanced Coordination Chemistry
*	Structural Inorganic Chemistry
*	Spectroscopy
*	English

She had successfully passed a written examination in these courses in partial fulfillment for the degree of Master of Science.

Approved
Vice - dean of Graduation studies



I do thank ALLAH for all gifts he gave to me. I wish to express my deep appreciation to prof. Dr. Marguerite A. wassef, Professor of inorganic Chemistry, Chemistry Department College for Women (Arts, Science and Education) Ain Shams University for her encouragement and moral support.

Words are no real assistance to express my deepest gratitude and thanks to Prof. Dr. Ali Mostafa A. Hassan, professor of inorganic Chemistry, faculty of science, AL-Azhar university for suggesting the point and for his Keen Supervision, fruitful assistance, useful discussion, his constructive guidance and inestimable encouragement he has given me in order to complete my thesis.

Also, I would like to express my sincere thank to Ass.prof. Dr. Hoda Abd-Elhakam bayoumi professor of inorganic Chemistry, Chemistry Department, Collage for Women (Arts, Science and Education) Ain Shams University for her encouragement, continuous help and careful guidance, throughout the accomplishment of this work.

I am indebted to Prof. Dr. Samia Mohtar Professor of Physical Chemistry and Head of Chemistry Department, University Collage for Women (Arts, Science and Education) Ain Shams University for her help and moral support.

Finally, my deep thanks to all staff members at my department for their encouragement and support.

Manara Ahmed



Ain shams University University College for women (Art, Science and Education) Cairo, Egypt.

# Synthesis and Characterization of Some New Schiff Base Complexes Using 1,6 - hexanediamine

A Thesis Submitted by

Manara Ahmed Mohammed Ayoub

for the Award of the M.Sc. Degree in Science (Inorganic and analytical Chemistry)

Supervised by

Prof. Dr. Marguerite A. wassef Prof. of Inorganic Chemistry University College for Women Ain shams university, Egypt Prof. Dr. Ali M. A. Hassan Prof. of Inorganic Chemistry Faculty of Science (Al-Azhar university), Egypt

Ass. Prof. Dr. Hoda A. Bayoumi Ass. Prof. of Inorganic Chemistry University College for Women Ain shams university, Egypt

Cairo - 2008



تخليق و توصيعت بعض المتراكبات الجديدة لقاعدة شيعت بأستخدام 6,1 ثنائبي الامين مكسان

مقدمة من

منارة أحمد محمد أيوب

للحصول علي درجة الماجيستير فيي العلوم

(كيمياء غيرالعضوية والتحليلية)

تدت اشراف

ا. د / مرجريت أنيس واصغت أستاذ الكيمياء غير العضوية كلية البنات – جامعة عين شمس

اد/ علي مصطفي على حسن أستاذ الكيمياء غير العضوية كلية العلوم- جامعة الازمر

حاله المحرب الحكم بير العضوية البنائد – جامعة غين همس كلية البنائد – جامعة غين همس (القاهرة 2008)

CONTENTS	
	Page
CONTENTS	I
LIST OF TABLES	V
LIST OF FIGURES	VIII
LIST OF SCHEMES	XV
AIM OF THE WORK	XVII
CHAPTER (I)	
INTRODUCTION	
General Background	1
Classification of The Schiff Base Compounds	4
(A) Literature Survey on Tetradentate Schiff Base	6
Ligands and Their Transition Metal Complexes	
(B) Applications of The Tetradentate Schiff Base	27
Complexes	
(C) Literature Survey on The Effect of Structure on	31
The Electrical Conductivity	
CHAPTER II	
EXPERIMENTAL	22
(A) Materials	33
(B) Synthesis of The Schiff Base Ligands	34
(i) Synthesis of N, N'- hexamethylenebis (Salicyliden-	34
amine)Schiff base H <sub>2</sub> L <sub>I</sub> (HBS)	
(ii) Synthesis of N, N'-hexamethylene bis (naphthalid-	35
enamine) Schiff base H <sub>2</sub> L <sub>II</sub> (HBN)	27
(C) Synthesis of Schiff Base Metal Complexes	37
(D) Quantitative Analysis of Metal Cations By	49
Gravimetric Methods	

(i) Determination of Chromium(III)	49
(ii) Determination of Manganese(II)	49
(iii) Determination of Iron(III) as Iron(III)Oxide	50
(iv) Determination of Cobalt(II)	50
(v) Determination of Ni(II)as nickel dimethyl- glyoximate	51
(vi) Determination of Copper(II)	51
(vii) Determination of Zinc(II)	52
(viii) Determination of Zr(IV) as ZrP <sub>2</sub> O <sub>7</sub>	52
(ix) Determination of Uranium(IV)	53
(E) Physical Measurements	54
(i) Elemental Analyses	54
(ii) FTIR Spectra	54
(iii) Mass Spectra	54
(iv) <sup>1</sup> H-NMR Spectra	55
(v) Ultraviolet and Visible Spectra	55
(vi) Thermo Gravimetric Analysis (TGA)	55
(vii) Magnetic measurements	55
(viii) Conductance Measurements	56
(ix) Electrical Conductivity Measurements	56

CHAPTER III RESULTS AND DISCUSSION	
(A) Schiff Base Ligand, H <sub>2</sub> L <sub>I</sub> (HBS).	59
(i) Infrared Spectrum of The Schiff Base, Ligand, H <sub>2</sub> L <sub>I</sub> (HBS).	59
(ii) Nuclear Magnetic Resonance Spectrum of The ligand H <sub>2</sub> L <sub>I</sub> (HBS).	65
(ii) Mass Spectrum of the ligand, H <sub>2</sub> L <sub>I</sub> (HBS).	70
(iv) UV Spectrum of the ligand, H <sub>2</sub> L <sub>I</sub> (HBS).	73
(B) Infrared Spectra of the Schiff Base Complexes of Ligand, H <sub>2</sub> L <sub>I</sub> (HBS).	76
(i) Infrared Spectra of Copper Complexes of Ligand H <sub>2</sub> L <sub>I</sub> (HBS) with Different Anions.	79
(ii) Infrared Spectra of the Metal Complexes of Ligand H <sub>2</sub> L <sub>I</sub> (HBS).	87
(iii) Nuclear Magnetic Resonance Spectrum of Zn(II) complex with H <sub>2</sub> L <sub>I</sub> (HBS)	95
(iv) Mass Spectra of the Metal Complexes of Ligand H <sub>2</sub> L <sub>I</sub> (HBS)	98
(v) Electronic Spectral Studies, Magnetic and Conductance of the Metal Complexes of Ligand H <sub>2</sub> L <sub>I</sub> (HBS).	104
(vi) Thermogravimetric Analysis of Metal Complexes of Ligand, H <sub>2</sub> L <sub>I</sub> (HBS).	120
(C) The Electrical Conductivity of The Metal Complexes of Ligand, H <sub>2</sub> L <sub>I</sub> (HBS).	126
(A) Schiff Base Ligand, H <sub>2</sub> L <sub>II</sub> (HBN).	135
(i) Infrared Spectrum of The Schiff Base Ligand, H <sub>2</sub> L <sub>II</sub> (HBN).	135
(ii) Nuclear Magnetic Resonance Spectrum of The ligand, H <sub>2</sub> L <sub>II</sub> (HBN).	141

(iii) Mass Spectrum of the ligand, H <sub>2</sub> L <sub>II</sub> (HBN).	144
(iv) UV Spectrum of the ligand, H <sub>2</sub> L <sub>II</sub> (HBN).	146
(B) Infrared Spectra of Metal Complexes of Schiff Base Ligand H <sub>2</sub> L <sub>II</sub> (HBN).	148
(i) Infrared Spectra of Copper Complexes of Ligand H <sub>2</sub> L <sub>II</sub> (HBN) with Different Anions.	148
(ii) Infrared Spectra of Metal Complexes of Ligand H <sub>2</sub> L <sub>II</sub> (HBN).	154
(iii) Nuclear Magnetic Resonance Spectra of Ni(II) and Zn(II) complexes with H <sub>2</sub> L <sub>II</sub> (HBN).	163
(iv) Mass Spectrum of Cr(III) Complex of Ligand H <sub>2</sub> L <sub>II</sub> (HBN).	169
(v) Electronic Spectral Studies, Magnetic and Conductance of Metal Complexes of Ligand H <sub>2</sub> L <sub>II</sub> (HBN).	172
(vi) Thermo gravimetric Analysis of Metal Complexes of Ligand H <sub>2</sub> L <sub>II</sub> (HBN).	189
(C) The Electrical Conductivity of The Metal Complexes of Ligand H <sub>2</sub> L <sub>II</sub> (HBN).	198
REFERENCES	209

## LIST OF TABLES

E 11 0 1		
Table 3.1.	Physical and analytical data of the Schiff	62
	base, H <sub>2</sub> L <sub>I</sub> , ligand and its copper metal	
	complexes.	
Table 3.2	Infrared frequencies (cm <sup>-1</sup> ) of Schiff base	63
	H <sub>2</sub> L <sub>I</sub> (HBS) ligand and their tentative	
	assignments.	
	usos grantos in	
Table 3.3.	The <sup>1</sup> H-NMR chemical shift (δ; ppm) of	68
10010 0101	Schiff base ligand, H <sub>2</sub> L <sub>I</sub> (HBS) in DMSO-	
	$d_6$ and its assignments.	
Table 3.4.	The electronic spectral data for Schiff base	75
1 4016 3.4.	-	13
Tolalo 2.5	ligand H <sub>2</sub> L <sub>I</sub> (HBS).	92
Table 3.5.	Characteristic infrared frequencies (cm <sup>-1</sup> )	82
	of the Schiff base ligand, H <sub>2</sub> L <sub>I</sub> (HBS) and	
	its copper metal complexes	
Table 3.6.	Physical and analytical data of the Schiff	90
	base ligand, $H_2L_I(HBS)$ and its transition	
	metal complexes	
Table 3.7.	Characteristic infrared frequencies (cm <sup>-1</sup> )	91
	of the Schiff base ligand, H <sub>2</sub> L <sub>I</sub> (HBS) and	
	its transition metal complexes.	
Table 3.8.	The <sup>1</sup> H-NMR chemical shift (δ; ppm) of	97
	the Zn(II) complex in in CF <sub>3</sub> COOD +	
	DMSO-d <sub>6</sub> and its assignments.	
Table 3.9.	Characteristic electronic transition bands,	110
1 4010 3.7.	magnetic moments and conductance of	110
	copper metal complexes, having different	
T.1.1. 0.10	anions, with Schiff base ligand, H <sub>2</sub> L <sub>I</sub> (HBS).	111
Table 3.10.	Characteristic electronic transition bands,	111
	magnetic moments and conductance of	
	transition metal complexes of Schiff base	
	ligand, $H_2L_I(HBS)$ .	