

قال تعالى : { اقْرَأْ بِاسْمِ رَبِّكَ الَّذِي خَلَقَ { 1 } خَلَـقَ { 1 } خَلَـقَ الْإِنسَـانَ مِـنْ عَلَـقٍ { 2 } اقْـرَأْ وَرَبُّـكَ الْأَكْـرَمُ { 3 } الْأَكْـرَمُ { 3 } الَّـذِي عَلَّـمَ بِـالْقَلَمِ { 4 } عَلَّـمَ الْإِنسَانَ مَا لَمْ يَعْلَمْ { 5 }

صدق الله العظيم

سورة العلق من الاية ( 1 – 5

i

## **DEDICATION**

To my father,

Mother,

Brothers,

Sisters,

Husband and children

## **ACKNOWLEDGMENTS**

I am greatly indebted to my supervisor Dr. Elmugdad Ahmed Ali who has supervised this study and offered his direction, encouragement and guidance throughout this wok, and without his patience, his invaluable and continuous assistance and advice. This work could not have become a reality, appreciate the assistance to Dr. Adam Khatir Sam, Dr. Abd Elrheem Mohamed, Dr. Noha Yousif, Dr. Abdella ElAabid and Ustaz. Ahmed Abker. My special thanks to the University of Bakht Al- Ruda for giving me this chance. I wish to record my deep gratefulness to the staff of chemical laboratories, College of Science, University of Sudan for Science and Technology, for their help and facilities kindly provided, specially Ibramim Khalifa, Ibrahim Yahya, Fathi, Adam and Eyhab.My greatest thanks to my teachers and Sudan University of Science and Technology, and staff of Al-Neelain University for their support.

#### **Abstract:**

Three hydroxamic acids (stearo - , oleo, plamatio) were prepared from stearic, oleic and palmitic acids by the reaction of their corresponding methyl esters with hydroxamine hydrochloride. The prepared hydroxamic acids were identified by their characteristic reaction colour with vanadium (V) (reddish-violet) and with iron (III) (blood – red), melting point, FT-IR. Spectra, <sup>1</sup>H-NMR, <sup>13</sup>C-NMR and elemental analysis. The extractive properties of the three hydroxamic acids using chloroform at pH 4, towards Mo(VI) were examined. Maximum extraction efficiencies of 92.92% for stearohydroxamic acid, 95.0% for oleohydroxamic acid and 93.0% for Palimitohydroxamic.

#### ملخص البحث

#### تم تحضير ثلاثة احماض هايدروكسيمية وهي :

استيارو هيدروكساميك - أوليو هيدروكساميك - بالميتو هيدروكساميك .

من حمض الاستاريك والاولياك والبلماتك بتفاعل اسراتها الميثلية مع هـ هيدروكسيل امين الحر .

تم التعرف على الاحطـض الهيدروكيمية التي تمـ تحضـرها عنـ طريقـ التفاعلات ذات الالوان المميزة مع الفانديوم (V) ( بنفسجي ) والحديد (III) ( أحمر) ، بقياس نقاط الانصهار ، أطياف الاشعة تحت الحمــاء ، الطنينـ النووي المغناطيسي $(^{13}C,^{1}H)$ ) وتحليل العناصر .

وجــد ان نسبــة المقـدـرة الاستخلاصيــة القصوــى لهـذـه الاحمـاـض الهيدروكسيمية الثلاث المذابة في الكلوروفورم عند الرقم الهايدروجيني 4 تجاه أيون للمولبيدنيوم (VI) كما يلي :

بلغت أعلى قيمة أستخلاصية لحامض الاستيارو هيدروكساميك للمولبيدنيوم . 92,92%، وبلغت اعلى قيمة استخلاصة لطمض اوليو هيدروكسميك للمولبيدنيوم 95% وكل بلغت اعلى قيمة استخلاصة لحامض بالميتوهيدروكساميك للمولبيدنيوم 93%.

#### **CONTENTS**

N.T.		
INO	Chapter One	page

1	Introduction	1
1.1	Hydroxamic acids	1
1.1.1	Classifications	2
1.1.2	Nomenclature	3
1.1.3	Structure	5
1.1.4	Preparation (Synthesis)	6
1.1.5	Characterization	8
1.1.6	Reactions of hydroxamic Acid	10
1.2	Extraction	13
1.2.1	Extraction of organic materials	13
1.2.2	Extraction of inorganic materials	14
1.2.3	Solvent extraction	15
1.2.4	Theory of the solvent extraction	16
1.2.4.1	Distribution law	16
1.2.5	Successive extraction	17
1.2.6	Separation of mixtures by extraction	19
1.2.7	Efficiency of extraction	19
1.2.8	An ideal solvent extraction	20
1.2.9	Factors Favoring solvent extraction	20
1.3	Analytical application of hydroxamic acid	23
1.3.1	Gravimetric determination of metals	23
1.3.2	Titrimetric application	25
1.3.3	Colorimetric application of hydroxamic acid	25
1.4	application of hydroxamic acid prepared in this	26
	department	
1.5	Chemistry of Molybdenum	31
1.5.1	Physical properties	32
1.5.2	Isotopes	33
1.5.3	Compounds	33
	Chapter two	

2	Equipment and reagents	36
2.1	preparation of stearohydroxamic acid	36
2.2	Preparation of palmitohydroxamic acids	38
2.3	Preparation of Oleohydroxamic acids	40
2.4	Extraction	42
2.4.1	Reagents	42
2.4.2	Buffer solutions.	43
2.4.3	Preparation of standard Calibration curve	43
2.4.4	Semi - quantitative test to evaluate the optimum pH	44
2.4.4.1	procedure of Stearohydroxamic acids (SHXA)	44
2.4.4.2	Procedure of Oleohydroxamic acids (OHXA)	45
2.4.4.3	Procedure of Palmitohydroxamic acids (PHXA)	45
2.5	Results	47
2.4.1	Extraction of Molybenum	49
	Chapter three	
	Discussion	54
	Appendix	<u> </u>
	Appendix ( 1 A ) I.R Spectrum of stearohydroxamic acid	59
	(1 A) I.R Spectrum of stearohydroxamic acid	59
	(1 A) I.R Spectrum of stearohydroxamic acid (1 B) <sup>1</sup> H- N.M.R Spectrum of stearohydroxamic acid	59 60
	(1 A) I.R Spectrum of stearohydroxamic acid (1 B) <sup>1</sup> H- N.M.R Spectrum of stearohydroxamic acid (1 C) <sup>13</sup> C-NMR Spectrum of Stearohydroxamic acid	59 60 61
	(1 A) I.R Spectrum of stearohydroxamic acid (1 B) <sup>1</sup> H- N.M.R Spectrum of stearohydroxamic acid (1 C) <sup>13</sup> C-NMR Spectrum of Stearohydroxamic acid (2 A) I.R Spectrum of palimtohydroxamic acid	59 60 61 62
	(1 A) I.R Spectrum of stearohydroxamic acid (1 B) <sup>1</sup> H- N.M.R Spectrum of stearohydroxamic acid (1 C) <sup>13</sup> C-NMR Spectrum of Stearohydroxamic acid (2 A) I.R Spectrum of palimtohydroxamic acid (2 B) <sup>1</sup> H- N.M.R Spectrum of palimtohydroxamic acid	59 60 61 62 63
	(1 A) I.R Spectrum of stearohydroxamic acid (1 B) <sup>1</sup> H- N.M.R Spectrum of stearohydroxamic acid (1 C) <sup>13</sup> C-NMR Spectrum of Stearohydroxamic acid (2 A) I.R Spectrum of palimtohydroxamic acid (2 B) <sup>1</sup> H- N.M.R Spectrum of palimtohydroxamic acid (2 C) <sup>13</sup> C- N.M.R Spectrum of palimtohydroxamic acid	59 60 61 62 63 64
	(1 A) I.R Spectrum of stearohydroxamic acid (1 B) <sup>1</sup> H- N.M.R Spectrum of stearohydroxamic acid (1 C) <sup>13</sup> C-NMR Spectrum of Stearohydroxamic acid (2 A) I.R Spectrum of palimtohydroxamic acid (2 B) <sup>1</sup> H- N.M.R Spectrum of palimtohydroxamic acid (2 C) <sup>13</sup> C- N.M.R Spectrum of palimtohydroxamic acid Appendix (3 A) I.R Spectrum of oleohydroxamic acid Appendix (3 B) <sup>1</sup> H- N.M.R Spectrum of oleohydroxamic	59 60 61 62 63 64 65
	(1 A) I.R Spectrum of stearohydroxamic acid (1 B) <sup>1</sup> H- N.M.R Spectrum of stearohydroxamic acid (1 C) <sup>13</sup> C-NMR Spectrum of Stearohydroxamic acid (2 A) I.R Spectrum of palimtohydroxamic acid (2 B) <sup>1</sup> H- N.M.R Spectrum of palimtohydroxamic acid (2 C) <sup>13</sup> C- N.M.R Spectrum of palimtohydroxamic acid Appendix (3 A) I.R Spectrum of oleohydroxamic acid Appendix (3 B) <sup>1</sup> H- N.M.R Spectrum of oleohydroxamic	59 60 61 62 63 64 65
	(1 A) I.R Spectrum of stearohydroxamic acid (1 B) <sup>1</sup> H- N.M.R Spectrum of stearohydroxamic acid (1 C) <sup>13</sup> C-NMR Spectrum of Stearohydroxamic acid (2 A) I.R Spectrum of palimtohydroxamic acid (2 B) <sup>1</sup> H- N.M.R Spectrum of palimtohydroxamic acid (2 C) <sup>13</sup> C- N.M.R Spectrum of palimtohydroxamic acid Appendix (3 A) I.R Spectrum of oleohydroxamic acid Appendix (3 B) <sup>1</sup> H- N.M.R Spectrum of oleohydroxamic	59 60 61 62 63 64 65 66

# Abbreviations and nomenclature Abbreviations

cm	Centimeter (10 <sup>-2</sup> of a meter )
dm	Decimeter (10 <sup>-1</sup> of a meter)
EDTA	Ethylene diamine tetra acetic acid
G	Gram
Mg	Milligram
Ppm	Parts per million
°C	degrees centigrade
u.v.	Ultra violet
i.r.	Infrared
n.m.r.	nuclear magnetic resonance
Nm	Nanometer (10 <sup>-9</sup> of a meter )
l l	Wavelength
Ł	Molar absorptivity
max.	Maximum
M	Morality
D	Density
Mol	Mole
A.R.	Analytical reagent
m.p.	Melting point
b.p.	Boiling point
M.Wt	Molecular weight
Conc.	Concentrated
Fig.	Figure
No.	Number
Lit.	Literature
A.	Appendix

## Nomenclature

SHXA	Stearohydroxamic acids
PHXA	Plamitohydroxamic acids
OHXA	Oleohydroxamic acids

## **List of Figures**

Fig (1)	Standard calibration curve for Mo (VI)	50
Fig (2)	Extraction curve showing distribution of Mo (VI) as	51

	function of pH for OHXA	
Fig (3)	Extraction curve showing distribution of Mo (VI) as	52
	function of pH for PHXA	
Fig(4)	Extraction curve showing distribution of Mo (VI) as	53
	function of pH for S <sub>st</sub> HXA	

## List of tables

Table (1.1)	Molybdenum compounds oxidation	33
Table (2.1)	Elemental analysis of SHXA	38
Table (2.2)	Elemental analysis of PHXA	40
Table (2.3)	Elemental analysis of OHXA	42
Table (2.4)	Results of standard calibration for Mo (VI).	47

Table (2.5)	Results of extraction of Mo (VI) with 0.25% w/v SHXA	47
	in chloroform.	
Table (2.6)	Results of extraction of Mo (VI) with 0.25% w/v OHXA	48
	in chloroform	
Table (2.7)	Results of extraction of Mo (VI) with 0.25% w/v PHXA	48
	in chloroform.	
Table (2.8)	Results of extraction of Mo (VI) with 0.25% w/v with	49
	three acids in chloroform.	
Table (3.1)	The infrared absorption frequencies (cm <sup>-1</sup> ) of hydroxamic	54
	acids prepared	
Table (3.2)	<sup>1</sup> H NMR spectral data of substituted hydroxamic acid	56-57
Table (3.3)	<sup>13</sup> C NMR data of substituted hydroxamic acid	57



# SUDAN UNIVERSITY OF SCIENCE AND TECHNOLOGY COLLEGE OF GRADUATE STUDIES

Solvent Extractive Properties of some Aliphatic Hydroxamic Acids towards Molybdenum (VI)

# A Thesis submitted for the fulfillment for the Degree Master of Science in Chemistry

By

#### **Maha Ibrahim Haroun**

B.Sc.(honor) – Bakht Al- Ruda University

### **Supervisor**

Dr. El mugdad Ahmed Ali

2013