

Chemistry Department Faculty of Science Ain Shams University

Extractive Studies for Separation and Preconcentration of Uranium(VI) and Thorium(IV) Ions from Liquid Samples prior their Determination

Thesis Submitted for Ph.D. Degree of Science in Chemistry

бу

Walaa Saeed Hafez Abd El-Latef

M.Sc. Chemistry (2014)
Assistant Lecturer -Nuclear Materials Authority

To

Chemistry Department
Faculty of Science
Ain Shams University
(2020)



Chemistry Department Faculty of Science Ain Shams University

Extractive Studies for Separation and Preconcentration of Uranium(VI) and Thorium(IV) Ions from Liquid Samples prior their Determination

Thesis Submitted for Ph.D. Degree of Science in Chemistry

by

Walaa Saeed Hafez Abd El-Latef

M.Sc. Chemistry (2014)

Under the Supervision of

Prof. Dr. Saad Abd El-Wahab Mohamed

Professor of Physical Chemistry, Chemistry Department, Faculty of Science, Ain Shams University

Prof. Dr. Tayseir Abd El-Samei Lasheen

Professor of Inorganic Chemistry, Nuclear Materials Authority

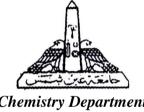
Dr. Dina Yeihia Sabry

Assistant Professor of Inorganic Chemistry, Chemistry Department, Faculty of Science, Ain Shams University

Dr. Ahmed Sami Helal

Lecturer of Inorganic Chemistry, Nuclear Materials Authority

(2020)



Chemistry Department Faculty of Science Ain Shams University

Approval Sheet

Thesis title: Extractive Studies for Separation and

Preconcentration of Uranium(VI) and Thorium(IV) Ions from Liquid Samples

prior their Determination

Name of candidate: Walaa Saeed Hafez Abd El-Latef

This thesis has been approved by:

Prof. Dr. Saad Abd El-Wahab Mohamed

Professor of Physical Chemistry, Chemistry Department, Faculty of Science, Ain Shams University

Prof. Dr. Tayseir Abd El-Samei Lasheen

Professor of Inorganic Chemistry, Nuclear Materials Authority

Dr. Dina Yeihia Sabry

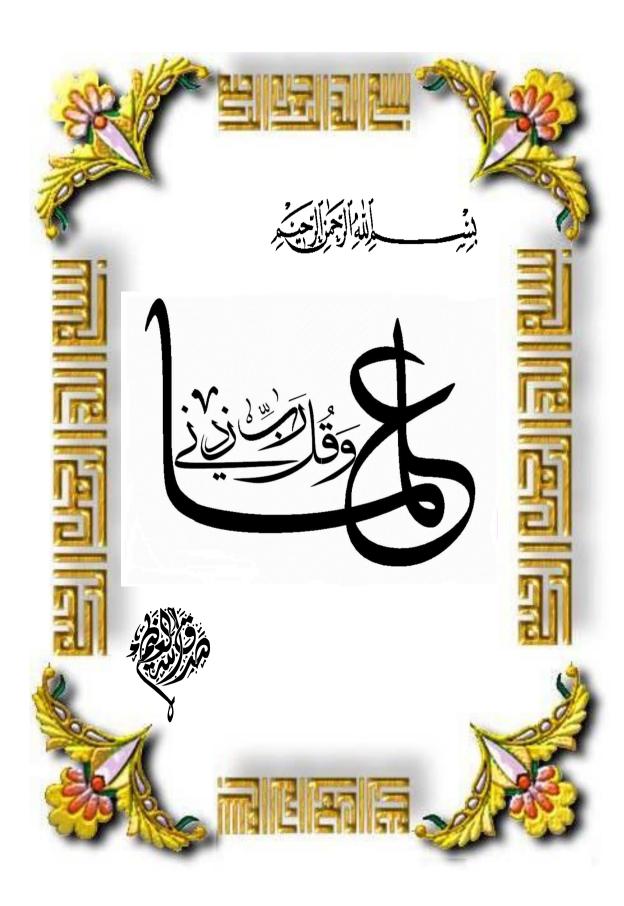
Assistant Professor of Inorganic Chemistry, Chemistry Department, Faculty of Science, Ain Shams University

Dr. Ahmed Sami Helal

Lecturer of Inorganic Chemistry, Nuclear Materials Authority

Head of Chemistry Department

Prof. Dr. Ayman Ayoub Abdel-Shafi



<u>ACKNOWLEDGMENT</u>

Firstly and foremost, my deep gratefulness, and thankful to "*ALLAH*" who gave me everything I have and the ability for completing this work.

I am indebted with sincere gratitude to *Prof. Dr. Saad A. Mohamed*, Professor of Physical Chemistry, Faculty of Science, Ain Shams University, for planning, reading and continuous encouragement and valuable advice are acknowledged with gratefulness.

I submit my highest appreciation to *Prof. Dr. Tayseir A. Lasheen*, Vice Head of Research Sector, Professor of Inorganic Chemistry, Nuclear Materials Authority, for supervision, her keen enthusiasm, sincere guidance, morale help and support, valuable suggestions and unrelenting efforts throughout each stage of this work.

Special thanks to *Dr. Dina Y. Sabry*, Assistant Professor of Inorganic Chemistry, Faculty of Science, Ain Shams University, for her continuous support and deep thanks to *Dr. Ahmed S. Helal*, Lecturer of Inorganic Chemistry, Nuclear Materials Authority, for encouragement.

My deepest appreciation and special thanks to *Dr. Mohamed A. Gado*, Assistant Professor of Chemistry, Nuclear Materials Authority, for his valuable guidance and continuous encouragement and I am grateful and thankful to *Prof. Dr. Hadi S. Gado*, Professor of Geology, Nuclear Materials Authority, for his reassurance and help.

In fact, I am truly thankful to *Prof. Dr. Mohammed G. El-Feky*, Head of Department of Geochemistry Exploration, Professor of Geochemistry, Nuclear Materials Authority, for his support and many thanks to *Dr. Neveen S. El-Sayed* for her morale help. I would like to thank my friends in Department of Isotopes Geology, Nuclear Materials Authority, especially *Dr. Marwa M. Rashad*

At the end, I am greatly indebted to my family; my mother, my brother, my daughters and other relatives, whose prayers and continuous moral support enabled me to achieve this hard task to be done.

CONTENTS

Object	Page
Object	1 age

CHAPTER 1

Introduction & literature review

1.1 Introduction	1
1.2. Literature review	7
1.2.1. Preconcentration and determination of thorium ions	7
1.2.1.1. Cloud point extraction procedure	7
1.2.1.2. Surfactant and micellar system	10
1.2.1.3. Types of surfactants	11
1.2.1.4. Principles of CPE complex formation	13
1.2.1.5. Cloud point extraction of thorium ions	
review	14
1.2.2. Separation and preconcentration of uranium ions	20
1.2.2.1. Basic principles of solid phase extraction	
procedure	21
1.2.2.2. Mechanism of retention of trace elements	24
1.2.2.3. Elution of trace elements from the sorbent	26
1.2.2.4. Solid phase extraction of uranium ions	
review	27

Object	Page

CHAPTER 2

Experimental

2.1. Chemicals and reagents	37
2.2. Solutions	37
2.2.1. Thorium(IV) and uranium(VI) standard solutions	37
2.2.2. Triton X-114 surfactant solution	37
2.2.3. Sodium chloride solution	37
2.2.4. 5-Br-PADAP solution	40
2.2.5. Complexing solution	40
2.2.6. Naphthalene solution	40
2.2.7. Sodium tetraphenyl borate solution	41
2.2.8. Buffer solutions preparation	41
2.2.8.1. Buffer solutions for thorium ions	41
2.2.8.2. Buffer solutions for uranium ions	41
2.2.9. Samples preparation	42
2.2.9.1. Rock samples dissolution procedure	42
2.2.9.2. Preparation of water samples	42
2.3. Recommended cloud point extraction procedure of thorium	
ions	43
2.4. Suggested solid phase extraction procedure of uranium	
ions	44

Object	Page
2.5. Preparation of naphthalene-NH ₄ -TPB adsorbent	44
2.6. Instrumentation	45
CHAPTER 3 Results & Discussion	
3.1. Cloud point extraction of thorium ions	47
3.1.1. Factors affecting on cloud point extraction of	
thorium ions	48
3.1.1.1. Absorption spectra	48
3.1.1.2. Effect of pH	48
3.1.1.3. Effect of buffer type	49
3.1.1.4. Effect of 5-Br-PADAP concentration	56
3.1.1.5. Effect of surfactant concentration	61
3.1.1.6. Effect of Triton X–114 volume	62
3.1.1.7. Effect of inorganic salts	65
3.1.1.8. Effect of equilibration temperature and	
incubation time	70
3.1.1.9. Effect of centrifugation time and rate	74
3.1.1.10. Effect of the order of addition of reagents	77
3.1.1.11. Effect of diluting agent	77

Object	Page
3.1.2. Interference studies	81
3.1.3. Analytical characteristics	84
3.1.4. Application	89
3.2. Solid phase extraction of uranium ions	95
3.2.1. Factors affecting on solid phase extraction of	
uranium ions	95
3.2.1.1. Absorption spectra	95
3.2.1.2. Effect of pH	97
3.2.1.3. Effect of buffer type	98
3.2.1.4. Effect of 5-Br-PADAP concentration	102
3.2.1.5. Effect of complexing solution	102
3.2.1.6. Effect of tetraphenyl borate concentration.	108
3.2.1.7. Effect of naphthalene volume	111
3.2.1.8. Effect of shaking time	111
3.2.1.9. Type of solvent	114
3.2.1.10. Effect of aqueous phase volume	114
3.2.2. Effect of foreign ions	119
3.2.3. Analytical performance	119
3.2.4. Retention capacity of the adsorbent	125
3.2.5. Scanning electron microscope characterization	125

Object	Page
3.2.6. Characterization studies	128
3.2.7. Application	131
Summary and conclusion	137
References	143
Arabic Summary	