







شبكة المعلومــات الجامعية التوثيق الالكتروني والميكروفيلم



جامعة عين شمس

التوثيق الالكتروني والميكروفيلم



نقسم بللله العظيم أن المادة التي تم توثيقها وتسجيلها علي هذه الأفلام قد اعدت دون آية تغيرات



يجب أن

تحفظ هذه الأفلام بعيداً عن الغبار

40-20 في درجة حرارة من 15-20 منوية ورطوبة نسبية من

To be kept away from dust in dry cool place of 15 – 25c and relative humidity 20-40 %











Solvent Extraction of Some Transition Metal Complexes With Some Organic Ligands

B1998

Using Some Organic Solvents

A Thesis Submitted

Вν

Shadia Mahmoud Mohamed Sirry

M.Sc. 1991

Faculty of Science, Aswan South Valley University

For

The Degree of Doctor of Philosophy

(1997)

Supervisors

Prof. Dr. R.M. Awadallah Professor of Analytical and Inorganic Chemistry

Dr. M.A. El Taher Lecturer of Physical Chemistry Dr. **A.H. Amrallah**Assistant Professor of

Analytical Chemistry

Dr. A.A. Gabr

Lecturer of Physical Chemistry

To
Chemistry Department
Faculty of Science, Aswan,
South Valley University
1417 - 1997

.

ACKNOWLEDGMENT

I wish to express my deepest thank and grateful gratitude to Prof. Dr. R.M. Awadallah Professor of Analytical and Inorganic Chemistry and Chairman of Chemistry Department, Faculty of Science, Aswan, South Valley University, for suggesting the plan of research, continuous supervision interpretation and discussion of the results as well as valuable revision.

I wish to express my deepest thank to Dr. A.M. Amrallah, Assistant Professor of Analytical Chemistry, for his supervision and facilities during the experimental work. I am also indepted to Dr. M.A. El Taher and Dr. A.A. Gabr, Lecturers of physical Chemistry for their supervision, help and encouregment during the experimental work.

I am grateful for the stuff members and colleagues of Chemistry Department, Faculty of Science, Aswan, South Valley University, for their assistance and cooperation.

S.M. Sirry

.

Solvent Extraction of Some Transition Metal Complexes With Some Organic Ligands Using Some Organic Solvents

Thesis Advisors:

Approved

Prof. Dr. R.M. Awadallah

Dr. A.H. Amrallah

Dr. M.A. El Taher

Dr. A.A. Gabr

Prof. Dr.

Chairman of Chemistry Department

Ţ

.

.

:

CONTENTS -

	page
Introduction	1
Aim of investigation	16
EXPERIMENTAL	
Synthesis of organic compounds	18
Solutions of metal ions	18
ligands' solutions	19
Solutions of masking agents	19
Solutions of other reagents	19
Buffer solutions	20
Organic solvents	20
Instruments	20
Working Procedurs	. /5
1. Effect of pH	21
2. Effect of ionic strength	21
3. Effect of solvent	22
4. Effect of shaking time	22
5. Effect of ligand concentration	23
6. Contiuous variation method	23
7. Successive extraction	24
8. Effect of metal ion concentration	24
9. Stripping procedure	25
10. Effect of interference	25
RESULTS AND DISCUSSION	
I - Extraction of Cu ²⁺ , Zn ²⁺ , Co ²⁺ and Ni ²⁺ with	27
thiazolylazoacetylacetone, TAA	
1. Effect of pH	28

28

2. Effect of shaking time	29
3. Effect of ionic strength .	29
4. Effect of solvent type	30
5. Effect of ligand concentration	31
6. Continous variation method	32
7. Validity of Beer's law	32
8. Effect of stripping agent	33
9. Effect of diverse ions	
a. Effect of diverse ions on the	34
determination of Cu ²⁴	
b. Effect of diverse ions on the	35
determination of Zn ²⁺	
c. Effect of diverse ions on the	35
determination of Co ²⁺	
d. Effect of diverse ions on the	36
determination of Ni ²⁺	
H- Extraction of Cu ²⁺ with o-hydroxyphenyl-	
azoacetylacetone HPAA	
L. Effect of pH	77
2. Effect of shaking time	78
3. Effect of ionic strength	79
4. Effect of types of solvent	79
5. Effect of ligand concentration	80
6. Continuous variation method	81
7. Validity of Beer's law	81
8. Effect of stripping agent	81
9. Effect of diverse ions	82
III- Extraction of Cu ²⁺ with o-hydroxyphenyl-	
azoethylacetoacetate, HPAEA	
L. Effect of pH	103
i. Caron or pri	1 (1.)

2. Effect of shaking time	104
3. Effect of ionc strength	104
4. Effect of types of solvents	104
5. Effect of ligand concentration	105
6. Continuous variation method	106
7. Validity of Beer's law .	106
8. Effect of stripping agents	107
9. Effect of diverse ions	107
IV- Evaluation of acid dissociation constants of ligands, extraction and formation constants of complexes and thermodynamic parameters	
 Evaluation of dissociation constants of 	
ligands	
a. Extraction-spectrophotometric method	127
b. Spectrophotometric method	128
i) Half height method	128
ii) Limiting absorbance method	129
 Evaluation of extraction constants of complexes 	140
 Evaluation of stability constants and free energy values of complexes 	141
SUMMARY	145
REFERENCES	150
SUMMARY IN ARABIC	

List of Tables

		Page
Table 1:	Characteristics of the synthesised organic ligands	26
Table 2:	Effect of pH on the extraction efficiency and distribution ratio of Cu ²⁺ - and Zn ²⁺ -thiazolylazoacetylacetonates	37
Table 3:	Effect of pH on the extraction efficiency and distribution ratio of Co ²⁺ - and Ni ²⁴ -thiazolylazoacetylacetonates	38
Table 4:	Effect of shaking time on the extraction efficiency of Cu ²⁺ -, Zn ²⁺ -, Co ²⁺ - and Ni ²⁺ -thiazolylazoacetylacetonate	39
Table 5;	Effect of ionic strength on the extraction efficiency of Cu ²⁺ -, Zn ²⁺ -, Co ²⁺ - and Ni ²⁺ -thiazolylazoacetylacetonate complexes	40
Table 6:	Effect of solvent on the extraction efficiency of Cu ²⁺ -, Zn ²⁺ -, Co ²⁺ - and Ni ²⁺ -complexes	41
Table 7:	Effect of ligand concentration on the extraction effeciency and distribution ratio of Cu ²⁺ - and Zn ²⁺ -thiazolylazo-acetylacetonate	42
Table 8:	Effect of ligand concentration on the extraction efficiency and distribution ratio of Co ²⁺ - and Ni ²⁺ -thiazolylazo-acetylacetonates	44
	"í V	