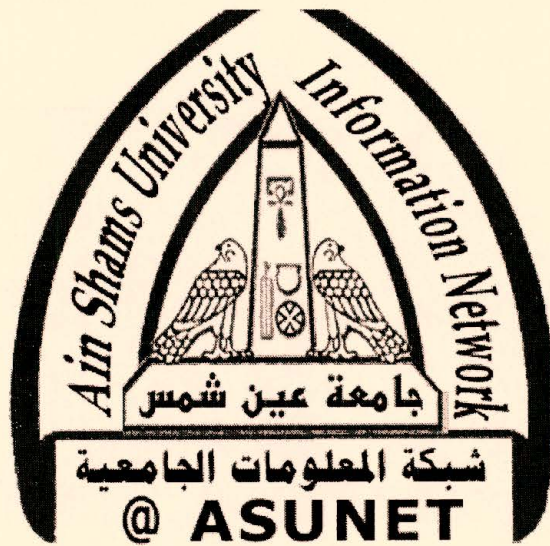




شبكة المعلومات الجامعية

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ





شبكة المعلومات الجامعية التوثيق الالكتروني والميكروفيلم



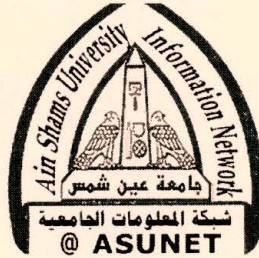
شبكة المعلومات الجامعية

جامعة عين شمس

التوثيق الالكتروني والميكروفيلم

قسم

نقسم بالله العظيم أن المادة التي تم توثيقها وتسجيلها
علي هذه الأفلام قد أعدت دون أية تغيرات



يجب أن

تحفظ هذه الأفلام بعيدا عن الغبار

في درجة حرارة من ١٥-٢٥ مئوية ورطوبة نسبية من ٢٠-٤٠%

To be Kept away from Dust in Dry Cool place of
15-25- c and relative humidity 20-40%



شبكة المعلومات الجامعية
@ASUNET

بالرسالة صفحات لم

ترد بالاصل



شبكة المعلومات الجامعية
@ ASUNET



ANALYTICAL STUDIES FOR ESTIMATING SOME RECENTLY ANTIBACTERIAL DRUGS

Dissertation submitted

By
Ibrahim Ahmed Hamed Agwa

For Doctoral Degree of Philosophy of Science (Ph.D) in
Chemistry (Analytical Chemistry)

Supervisors

Professor Doctor

Hassan Ali Dessouki

Prof. of Inorganic and
Analytical Chemistry and
Head of Chemistry
Department

Professor Doctor

Alaa El-Sayed Amin

Prof. of Analytical
Chemistry

Benha University
Faculty of Science
Chemistry Department

2006



B

01-0



ANALYTICAL STUDIES FOR ESTIMATING SOME RECENTLY ANTIBACTERIAL DRUGS

Dissertation submitted

By
Ibrahim Ahmed Hamed Agwa

For Doctoral Degree of Philosophy of Science (Ph.D) in
Chemistry (Analytical Chemistry)

Supervisors

Professor Doctor

Hassan Ali Dessouki

Prof. of Inorganic and
Analytical Chemistry and
Head of Chemistry
Department

Professor Doctor

Alaa El-Sayed Amin

Prof. of Analytical
Chemistry

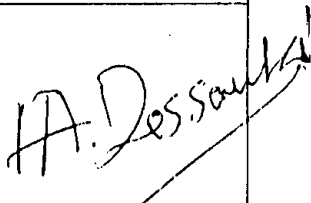
Benha University
Faculty of Science
Chemistry Department

2006

(Supervisors)

Entitled: ANALYTICAL STUDIES FOR ESTIMATING SOME RECENTLY ANTIBACTERIAL
DRUGS

Presented by: Ibrahim Ahmed Hamed Agwa

No.	Name	Position	Signature
1-	Prof. Dr. Hassan Ali Dessouki	Prof. of Inorganic and Analytical Chemistry and Head of Chemistry Department, Faculty of science, Benha University	
2-	Prof. Dr. Alaa El-Sayed Amin	Prof. of Analytical Chemistry, Faculty of science, Benha University	

(Approval Sheet)

**Entitled: ANALYTICAL STUDIES FOR ESTIMATING SOME RECENTLY
ANTIBACTERIAL DRUGS**

Presented by: Ibrahim Ahmed Hamed Agwa

No.	Name	Position	Signature
1-			
2-			
3-			
4-			

Date of discussion: / /

Dean of college agreement.

ACKNOWLEDGMENT

I am deeply thankful to Allah by the grace whom the progress and success of this work possible.

I am also indebted with sincere gratitude to **Prof. Dr.Hassan Ali Dessouki**, Professor of Inorganic and Analytical Chemistry and Head of Chemistry Department, Faculty of Science, Benha University, for problem solvents, critical continuous supervision and help during the work, discussion and help during the scope of this work and in preparing the thesis.

My great indebtedness and sincere gratitude to **Prof. Dr. Alaa Al-sayed Amin**, Professor of Analytical Chemistry, Faculty of Science, Benha University, for critical continuous supervision and help during the work, discussion and help during the scope of this work and in preparing the thesis.

My thanks to all staff members in Chemistry Department in EIPICO (Egyptian International Pharmaceutical Industries Corporation) for their help.

My thanks to all staff members in Chemistry Department in Faculty of Science Benha University for their help.

Ibrahim Ahmed Hamed Agwa

Contents

CHAPTER I

1. Introduction	1
1.1 High performance liquid chromatographic methods	2
1.2 Spectrophotometric methods.....	34

CHAPTER II

2. Experimental.....	51
2.1 Materials and Methods for HPLC.....	51
2.1.1 Apparatus.....	51
2.1.2 Stationary Phases.....	51
2.1.3 Chemical Reagents	51
2.1.4 Samples.....	52
2.1.5 Standards and Samples Preparations	52
2.1.5.1 Standards Preparations.....	52
2.1.5.2 Samples Preparations.....	53
2.1.5.3.1 Tablets	53
2.1.5.3.2 Injections	53
2.1.6 Mobile Phases.....	54
2.1.7 Pharmacopial method for ofloxacin	55
2.2 For spectral measurement.....	56
2.2.1 Apparatus.....	56
2.2.2 Drugs	57
2.2.3 Reagents.....	57
2.2.4 Determination of the molecular structure.....	57
2.2.5 Spectrophotometric determination of the drug under investigation using ion-pair complex formation with acid	

dyes.....	58
2.2.5.1 Procedures for the determination of the studied drugs in authentic powder using Reagent I.....	58
2.2.5.2 Procedures for the determination of the studied drugs in authentic powder using Reagent II.....	59
2.2.5.3 Procedures for the determination of the studied drugs in authentic powder using Reagent III.....	59
2.2.5.4 Procedures for the determination of the studied drugs in authentic powder using Reagent IV.....	59
2.2.5.5 Procedures for the determination of the studied drugs in authentic powder using Reagent V.....	60
2.2.6 Procedure for dosage forms	60
2.2.6.1 For tablets.....	60
2.2.6.2 For Injection and eye drops.....	61

CHAPTER III

3. Results and discussion.....	62
3.1 High-performance liquid chromatography.....	62
3.1.1 Theoretical bases.....	62
3.1.1.1 Parameters of chromatographic separation.....	62
3.1.1.2 Resolution, R_s	63
3.1.1.3 Capacity factor, k'	64
3.1.1.4 Selectivity, α	65
3.1.1.5 Tailing factor, T	65

3.1.1.6	Relative standard deviation	66
3.1.1.7	Efficiency of column, N	66
3.1.1.8	Band broadening	68
3.1.1.9	Silica-bonded-phase supports and mobile phases	70
3.1.1.10	Normal- and reversed-phase chromatography ...	71
3.1.2	Enrofloxacin	73
3.1.2.1	System Suitability Test	73
3.1.2.2	Method validation.....	75
3.1.2.2.1	Specificity	75
3.1.2.2.2	Linearity and range	79
3.1.2.2.3	Limit of detection	81
3.1.2.2.4	Limit of quantitation.....	81
3.1.2.2.5	Accuracy (Recovery).....	82
3.1.2.2.6	Precision.....	85
3.1.2.3	Determination of enrofloxacin antibacterial component in pharmaceutical formulations ...	87
3.1.2.4	Conclusion	88
3.1.3	Lomefloxacin	89
3.1.3.1	System Suitability Test	89
3.1.3.2	Method validation.....	91
3.1.3.2.1	Specificity	91
3.1.3.2.2	Linearity and range	95
3.1.3.2.3	Limit of detection	97
3.1.3.2.4	Limit of quantitation.....	97
3.1.3.2.5	Accuracy (Recovery).....	98
3.1.3.2.6	Precision.....	101
3.1.3.3	Determination of lomefloxacin antibacterial component in pharmaceutical formulations ...	103
3.1.3.4	Conclusion	104
3.1.4	Ofloxacin	105

3.1.4.1	System Suitability Test	105
3.1.4.2	Method validation.....	107
3.1.4.2.1	Specificity	107
3.1.4.2.2	Linearity and range	111
3.1.4.2.3	Limit of detection	113
3.1.4.2.4	Limit of quantitation.....	113
3.1.4.2.5	Accuracy (Recovery).....	114
3.1.4.2.6	Precision.....	117
3.1.4.3	Determination of ofloxacin antibacterial component in pharmaceutical formulations ...	120
3.1.4.4	Conclusion	120
3.2	Spectrophotometric Studies	122
3.2.1	Determination of the studied drugs by complex formation with acid dyes	122
3.2.1.1	Absorption spectra of the studied drugs using reagent I	122
3.2.1.1.1	Effect of pH.....	122
3.2.1.1.2	Effect of shaking time	122
3.2.1.1.3	Effect of the extracting solvent	123
3.2.1.1.4	Effect of reagent concentration	123
3.2.1.1.5	Molecular ratio of complexes.....	123
3.2.1.1.6	Sequence of addition	124
3.2.1.1.7	Suggested mechanism	124
3.2.1.1.8	Evaluation of stability constants of the ion-pair complexes	126
3.2.1.1.9	Statistical analysis.....	126
3.2.1.1.10	Validity of Beer's law	128
3.2.1.1.11	Accuracy and precision	129
3.2.1.1.12	Analytical applications	128
3.2.1.2	Absorption spectra of the studied drugs using reagent II.....	141

3.2.1.2.1	Effect of pH.....	141
3.2.1.2.2	Effect of shaking time	141
3.2.1.2.3	Effect of the extracting solvent	141
3.2.1.2.4	Effect of reagent concentration	142
3.2.1.2.5	Molecular ratio of complexes.....	142
3.2.1.2.6	Sequence of addition	142
3.2.1.2.7	Suggested mechanism	143
3.2.1.2.8	Evaluation of stability constants of the ion-pair complexes	144
3.2.1.2.9	Validity of Beer's law	144
3.2.1.2.10	Accuracy and precision	145
3.2.1.2.11	Analytical applications	145
3.2.1.3	Absorption spectra of the studied drugs using reagent III.....	156
3.2.1.3.1	Effect of pH.....	156
3.2.1.3.2	Effect of shaking time	156
3.2.1.3.3	Effect of the extracting solvent	156
3.2.1.3.4	Effect of reagent concentration	157
3.2.1.3.5	Molecular ratio of complexes.....	157
3.2.1.3.6	Sequence of addition	157
3.2.1.3.7	Suggested mechanism	158
3.2.1.3.8	Evaluation of stability constants of the ion-pair complexes	159
3.2.1.3.9	Validity of Beer's law	159
3.2.1.3.10	Accuracy and precision	160
3.2.1.3.11	Analytical applications	160
3.2.1.4	Absorption spectra of the studied drugs using reagent IV.....	172
3.2.1.4.1	Effect of pH.....	172
3.2.1.4.2	Effect of shaking time	172
3.2.1.4.3	Effect of the extracting solvent	172