



# UNIVERSITÀ DEGLI STUDI DI PALERMO

<b>DEPARTMENT</b>	Scienze e Tecnologie Biologiche, Chimiche e Farmaceutiche
<b>ACADEMIC YEAR</b>	2018/2019
<b>MASTER'S DEGREE (MSC)</b>	PHARMACEUTICAL CHEMISTRY AND TECHNOLOGIES
<b>SUBJECT</b>	DRUG ANALYSIS
<b>TYPE OF EDUCATIONAL ACTIVITY</b>	B
<b>AMBIT</b>	50323-Discipline Chimiche, Farmaceutiche e Tecnologiche
<b>CODE</b>	01205
<b>SCIENTIFIC SECTOR(S)</b>	CHIM/08
<b>HEAD PROFESSOR(S)</b>	CASCIOFERRO STELLA Professore Associato Univ. di PALERMO MARIA PARRINO BARBARA Professore Associato Univ. di PALERMO
<b>OTHER PROFESSOR(S)</b>	
<b>CREDITS</b>	10
<b>INDIVIDUAL STUDY (Hrs)</b>	156
<b>COURSE ACTIVITY (Hrs)</b>	94
<b>PROPAEDEUTICAL SUBJECTS</b>	01799 - ANALYTICAL CHEMISTRY 01211 - PHARMACEUTICAL ANALYSIS OF DRUGS
<b>MUTUALIZATION</b>	
<b>YEAR</b>	3
<b>TERM (SEMESTER)</b>	2° semester
<b>ATTENDANCE</b>	Mandatory
<b>EVALUATION</b>	Out of 30
<b>TEACHER OFFICE HOURS</b>	<b>CASCIOFERRO STELLA MARIA</b> Monday 12:30 14:30 Ufficio del docente Wednesday 12:30 14:30 Ufficio del docente  <b>PARRINO BARBARA</b> Monday 13:00 14:00 Viale delle Scienze, Ed. 16, piano terra Thursday 13:30 14:30 Viale delle Scienze, Ed. 16, piano terra Friday 10:00 12:00 Viale delle Scienze, Ed. 16, piano terra

DOCENTE: Prof.ssa BARBARA PARRINO- *Lettere M-Z*

<b>PREREQUISITES</b>	Organic Chemistry, Analytical Chemistry
<b>LEARNING OUTCOMES</b>	<p>Knowledge and Understanding Acquisition of methods and tools for quality control. Capacity to describe the problems involved in the quantitative determinations.</p> <p>Professional Skills Capacity to recognize and apply independently, methodologies needed for the assay of drugs registered in the Italian Official Pharmacopoeia.</p> <p>Intellectual Skills Be able to resolve problems designed to evaluate the level of knowledge acquired on the topics and techniques that are the object of the lectures and laboratory practice.</p> <p>Transferable Skills Ability to explain the problems and the calculations useful for determining the purity degree of a compound.</p> <p>Follow on Skills Ability to use the knowledge acquired during the course in order to resolve new analytical problems</p>
<b>ASSESSMENT METHODS</b>	<p>Oral examination. Student must verbally answer at least three questions about the topics of the program, with reference to the recommended texts. The examination assessment will be considered based on the level of knowledge, the properties of language as well as the clarity of the topics covered.</p> <p>The score is expressed using a 30-point scale (from min 18/30 to max 30/30 cum laude) The student gains a minimum range score (18-20/30) after showing general knowledge and comprehension of the treated topics, and expressing them with proper scientific vocabulary, even if not adequately articulated. The score will be increased (range score from 20/30 to 28/30) the more the candidate shows a deep knowledge of the topics, coming both from the information he acquired during the course and from a precise and deep personal study of the recommended texts, and also if he shows autonomy of judgement and comprehension of the applicable properties of the newly acquired knowledge. Positive scores will also be given to a clear and articulated presentation, along with the correct use of scientific vocabulary. The score of 30/30 and 30/30 cum laude will be gained by the candidate who shows optimal knowledge of the topics, which he expresses in a clear and articulated way with optimal language skills and good analytical skills, showing his judgement autonomy and his application ability of the newly acquired knowledge.</p>
<b>EDUCATIONAL OBJECTIVES</b>	The training objective of the course is to make the student able to acquire the basic skills needed to resolve analytical problems of drugs registered in Italian F.U
<b>TEACHING METHODS</b>	Frontal lectures and individual laboratory practices
<b>SUGGESTED BIBLIOGRAPHY</b>	<p>G.C.Porretta: "Analisi Quantitativa di Composti Farmaceutici"; CISU D.C.Harris: "Chimica Analitica Quantitativa"; Zanichelli Skoog West: "Chimica Analitica"; Beckett Stenlake: "Practical Pharmaceutical Chemistry</p>

## SYLLABUS

Hrs	Frontal teaching
4	The drug from synthesis to market. Sources of impurities in pharmaceuticals. Guide to the use of the Italian Official Pharmacopoeia IX Ed. Chemical purity, its control, impurity sources, physical, chemical-physical and chemical methods
10	Analytical procedure. Gravimetric analysis. Characteristics of gravimetric precipitates. Filterability and purity of the precipitates. Colloidal precipitates. Crystalline precipitates. Precipitating reagents in the homogeneous phase. Drying and incineration of the precipitates. Applications of Gravimetric Analysis. Calculation of the results in gravimetric analysis. Gravimetric factor. Gravimetric assays of substances listed in F.U. IX Ed.
4	Methods of extraction. Introduction to volumetric analysis. Requirements of a volumetric reaction. Primary standard. Standard solution. Equivalent point and end point. Titration error. Titration curves. Parameters that influence the titration curves.
6	Titration curves of precipitation reactions. Determination of the end point. Analytical application of the precipitation titration (Mohr, Volhard, Fajans). Precipitimetric assays of substances listed in F.U. IX Ed.
12	Acid-base titrations. Indicators. Titration curves. Titrations of strong acids with strong bases. pH of a weak acid, hydrolysis, buffer. Titrations of weak monoprotic acids with strong bases. Titrations of monoprotic weak bases with strong acids. Titration of polyprotic acids. Titrations of polyprotic bases. Titrations of acid and base mixtures. Acidimetric dosage of substances listed in F.U. IX Ed. Alcalimetric dosage of substances listed in F.U. IX Ed
8	Non-aqueous solvents: Solvents classification. Intrinsic acidity and basicity. Autoprotolysis constant. Leveling effect. Weak acids and bases in amphiprotic solvents. The choice of the solvent for a titration in non-aqueous environment. Examples of dosages in non-aqueous solvents of substances listed in F.U. IX Ed.

## SYLLABUS

Hrs	Frontal teaching
6	Complexometry. Chelate complexes formation. Stability constant of the complexes. Ethylenediaminetetraacetic acid (EDTA). Influence of pH on the stability of the complexes with EDTA. Titration curves with EDTA. Indicators for titration with EDTA. Types of complexometric titrations: direct titration, displacement, indirect, alcalimetric. Selectivity in complexometric titrations. Permanent and temporary hardness. Some complexometric dosages of substances listed in F.U.IX Ed
8	Electrochemistry principles . Electrochemical cells. Nernst equation. Influence of pH in redox reactions. Influence of precipitating and complexing agents on redox potentials. Calculation of the equilibrium constants of a reaction. Ossidimetric assays of substances listed in the F.U. IX Ed. Titration curves of redox reaction. Redox indicators. Volumetric oxidizing: permanganometry, Iodometry, Iodimetry, Bromometry, cerimetry. Ossidimetric assays of substances listed in F.U. IX Ed.
6	Principle of the potentiometer, Types of electrodes. Potentiometric titration, determination of the end point. Definition and conductometric sizes. Measurements of conductivity. Conductometric cells. Conductometric titrations : strong acid-strong base, weak base-strong acid, acid, precipitation reactions. Potentiometric determinations in F.U.
Hrs	Workshops
30	Volume flow rate, pipetted, zeroing. Chlorides (Mohr, Fajans). Chlorides (Vohard) Acid / Base-phenolphthalein. Acid / Base-methylorange. Mixture carbonates / bicarbonates. Mixture of acids. Chloral hydrate. Hardness. Determination hydrogen peroxide.Vitamin C. Chloramine T. Phenol. acid / base potentiometric. acid / base conductometric

DOCENTE: Prof.ssa STELLA MARIA CASCIOFERRO- *Lettere A-L*

<b>PREREQUISITES</b>	Organic Chemistry, Analytical Chemistry
<b>LEARNING OUTCOMES</b>	<p>Knowledge and Understanding Acquisition of methods and tools for quality control. Capacity to describe the problems involved in the quantitative determinations.</p> <p>Professional Skills Capacity to recognize and apply independently, methodologies needed for the assay of drugs registered in the Italian Official Pharmacopoeia.</p> <p>Intellectual Skills Be able to resolve problems designed to evaluate the level of knowledge acquired on the topics and techniques that are the object of the lectures and laboratory practice.</p> <p>Transferable Skills Ability to explain the problems and the calculations useful for determining the purity degree of a compound.</p> <p>Follow on Skills Ability to use the knowledge acquired during the course in order to resolve new analytical problems</p>
<b>ASSESSMENT METHODS</b>	<p>Oral examination. Student must verbally answer at least three questions about the topics of the program, with reference to the recommended texts. The examination assessment will be considered based on the level of knowledge, the properties of language as well as the clarity of the topics covered.</p> <p>The score is expressed using a 30-point scale (from min 18/30 to max 30/30 cum laude) The student gains a minimum range score (18-20/30) after showing general knowledge and comprehension of the treated topics, and expressing them with proper scientific vocabulary, even if not adequately articulated. The score will be increased (range score from 20/30 to 28/30) the more the candidate shows a deep knowledge of the topics, coming both from the information he acquired during the course and from a precise and deep personal study of the recommended texts, and also if he shows autonomy of judgement and comprehension of the applicable properties of the newly acquired knowledge. Positive scores will also be given to a clear and articulated presentation, along with the correct use of scientific vocabulary. The score of 30/30 and 30/30 cum laude will be gained by the candidate who shows optimal knowledge of the topics, which he expresses in a clear and articulated way with optimal language skills and good analytical skills, showing his judgement autonomy and his application ability of the newly acquired knowledge.</p>
<b>EDUCATIONAL OBJECTIVES</b>	The training objective of the course is to make the student able to acquire the basic skills needed to resolve analytical problems of drugs registered in Italian F.U
<b>TEACHING METHODS</b>	Frontal lectures and individual laboratory practice
<b>SUGGESTED BIBLIOGRAPHY</b>	<p>G.C.Porretta: "Analisi Quantitativa di Composti Farmaceutici"; CISU D.C.Harris: "Chimica Analitica Quantitativa"; Zanichelli Skoog West: "Chimica Analitica"; Beckett Stenlake: "Practical Pharmaceutical Chemistry</p>

## SYLLABUS

Hrs	Frontal teaching
4	The drug from synthesis to market. Sources of impurities in pharmaceuticals. Guide to the use of the Italian Official Pharmacopoeia IX Ed. Chemical purity, its control, impurity sources, physical, chemical-physical and chemical methods.
10	Analytical procedure. Gravimetric analysis. Characteristics of gravimetric precipitates. Filterability and purity of the precipitates. Colloidal precipitates. Crystalline precipitates. Precipitating reagents in the homogeneous phase. Drying and incineration of the precipitates. Applications of Gravimetric Analysis. Calculation of the results in gravimetric analysis. Gravimetric factor. Gravimetric assays of substances listed in F.U. IX Ed.
4	Methods of extraction. Introduction to volumetric analysis. Requirements of a volumetric reaction. Primary standard. Standard solution. Equivalent point and end point. Titration error. Titration curves. Parameters that influence the titration curves.
6	Titration curves of precipitation reactions. Determination of the end point. Analytical application of the precipitation titration (Mohr, Volhard, Fajans). Precipitometric assays of substances listed in F.U. IX Ed.
12	Acid-base titrations. Indicators. Titration curves. Titrations of strong acids with strong bases. pH of a weak acid, hydrolysis, buffer. Titrations of weak monoprotic acids with strong bases. Titrations of monoprotic weak bases with strong acids. Titration of polyprotic acids. Titrations of polyprotic bases. Titrations of acid and base mixtures. Acidimetric dosage of substances listed in F.U. IX Ed. Alcalimetric dosage of substances listed in F.U. IX Ed
8	Non-aqueous solvents: Solvents classification. Intrinsic acidity and basicity. Autoprotolysis constant. Leveling effect. Weak acids and bases in amphiprotic solvents. The choice of the solvent for a titration in non-aqueous environment. Examples of dosages in non-aqueous solvents of substances listed in F.U. IX Ed.

## SYLLABUS

Hrs	Frontal teaching
6	Complexometry. Chelate complexes formation. Stability constant of the complexes. Ethylenediaminetetraacetic acid (EDTA). Influence of pH on the stability of the complexes with EDTA. Titration curves with EDTA. Indicators for titration with EDTA. Types of complexometric titrations: direct titration, displacement, indirect, alcalimetric. Selectivity in complexometric titrations. Permanent and temporary hardness. Some complexometric dosages of substances listed in F.U.IX Ed
8	Electrochemistry principles . Electrochemical cells. Nernst equation. Influence of pH in redox reactions. Influence of precipitating and complexing agents on redox potentials. Calculation of the equilibrium constants of a reaction. Ossidimetric assays of substances listed in the F.U. IX Ed. Titration curves of redox reaction. Redox indicators. Volumetric oxidizing: permanganometry, Iodometry, Iodimetry, Bromometry, cerimetry. Ossidimetric assays of substances listed in F.U. IX Ed.
6	Principle of the potentiometer, Types of electrodes. Potentiometric titration, determination of the end point. Definition and conductometric sizes. Measurements of conductivity. Conductometric cells. Conductometric titrations : strong acid-strong base, weak base-strong acid, acid, precipitation reactions. Potentiometric determinations in F.U.
Hrs	Workshops
30	Volume flow rate, pipetted, zeroing. Chlorides (Mohr, Fajans). Chlorides (Vohard) Acid / Base-phenolphthalein. Acid / Base-methylorange. Mixture carbonates / bicarbonates. Mixture of acids. Chloral hydrate. Hardness. Determination hydrogen peroxide.Vitamin C. Chloramine T. Phenol. acid / base potentiometric. acid / base conductometric